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Designing New Aluminium Alloy by Orthogonal Array Analysis

Student Name: ZI XUAN, LIM

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Supervisor: Prof. MING XING, ZHANG

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Faculty of Engineering, Architecture and Information Technology

Abstract: The type of alloying elements and its respective percentages is important to the overall mechanical properties of aluminium alloy. The primary purpose of this thesis is designing new aluminium alloy which has better hardness compared to commercially available ones in the market. Constituents and weight of aluminium alloy will be determined by statistical modelling known as Taguchi's Method Orthogonal Array. Aluminium alloys were casted by permanent mould casting and heat treated at optimum heat treatment temperature and time to achieve peak hardness. A total of 16 experimental runs was produced by orthogonal array modelling. This thesis will mostly document the result of experimental run 7, 8 and 9. Analysis of aluminium alloy will be done by Vickers Hardness Test and microstructure analysis under optical microscope. The results obtained from experimental run 7, 8 and 9 will be correlated with other experimental run result conducted by my teammates to determine the alloy which yields the highest hardness and alloying elements and percentage which will produce aluminium alloy with high hardness through orthogonal array analysis. Hardness result of each alloy mostly shows above average hardness compared to commercially available ones at 140 HV. The highest recorded hardness obtained is alloy 8 at 240 HV. Through analysis of result, it is established that alloys with silicon, nickel, copper and magnesium show the highest average hardness. For following iteration (future casting and research), a general optimum alloying percentage is given and with a general emphasis on these 4 alloying elements. For commercialisation though, more work must be done based on the findings and results of this thesis. Designing new alloys requires multiple iterations and usually can't be done with one research. Future iteration recommendation includes optimising the casting process, heat treatment process and altering the alloying constituents based on the orthogonal analysis results.

Table of Contents

1. INTRODUCTIONS 1

1.1 Historical Advancement and Motivation of Alloy Development 2

2. OBJECTIVES 3

3. LITERATURE REVIEWS 5

3.1 Aluminium Alloy Metallurgy, Alloy and Alloying Elements 5

3.1.1 Aluminium Alloying Elements, Grain Refiner and Respective Effects 6

3.2 Modern Method to Design New Alloy 10

3.2.1 Design Of Experiment(DOE) 11

3.2.2 Taguchi's Method Orthogonal Array 12

3.3 Casting Metal 15

3.3.1 Permanent Mould Casting and Justification 16

3.3.2 Aluminium Casting Fluidity 18

3.3.3 Cooling and Solidifications 19

3.4 Heat Treatment 21

3.4.1 Solution Heat Treatment 21

3.4.2 Quenching 24

3.4.3 Age Hardening 25

3.5 Microstructure of Aluminium Alloy and Microstructure Analysis 27

3.5.1 Microstructure of Aluminium Alloy 27

3.5.2 Microstructure Analysis 28

3.6 Hardness and Hardness Test 30

4. METHODOLOGY 33

4.1 Material and Preparation 33

4.2 Constructing Orthogonal Array 33

4.3 Casting Process 35

4.4 Methodology and Justification of Heat Treatment Process 36
4.4.1 Solution Heat Treatment Process 38
4.4.2 Age Hardening Process 39
4.5 Vickers's Hardness Test Methodology 39
4.6 Microstructure Analysis Methodology and Reasoning 40
4.7 Analysis by Orthogonal Array 41
5. RESULTS 42
5.1 Sample 7: Al-4Si-6Cu-2Mg-2Zn-0.6Mn-1Sn-2Ni-1Ti 42
5.1.1 Sample 7: Casted Alloy and Microstructure 42
5.1.2 Sample 7: Solution Treatment Results 45
5.1.3 Sample 7 Aging Curve and Aged Hardness Results 46
5.2 Sample 8: Al-6Si-4Cu-4Mg-6Zn-0.2Mn-4Cr-6Ni-1Ti 48
5.2.1 Sample 8: Casted Alloy and Microstructure 48
5.2.2 Sample 8: Solution Treatment Results 50
5.2.3 Sample 8: Aging Curve and Aged Hardness Results 52
5.3 Sample 9: Al-4Cu-6Mg-4Zn-0.2Mn-4Ni-1Ti 54
5.3.1 Sample 9: Casted Alloy and Microstructure 54
5.3.2 Sample 9: Solution Treatment Results 57
5.3.3 Sample 9: Aging Curve and Aged Hardness Results 58
5.4 Orthogonal Array Analysis Result 60
6. DISCUSSION 65
6.1 Future Research 68
7. CONCLUSIONS 69
8. REFERENCES 71
9. APPENDIX 79

Table 1: Aluminium alloying element effect and details. [26] [27] [28] 9
Table 2: Design of experiments and its area of factor. 12
Table 3: Steps to conducting Taguchi's orthogonal array. 14
Table 4: Details of semi-permanent and permanent mould casting. 17
Table 5: Shows commonly used quenching media and description. [18] 24
Table 6: Preparatory steps for microstructure analysis. [19] 28
Table 7: Details the advantages and disadvantages of Vickers pyramid hardness test. [10] 32
Table 8: Allowable percentage of alloying to be added for to aluminium alloy. 33
Table 9: Shows all the factor level assigned to the columns. 34
Table 10: Shows the influence value of each factor level of a variable. 35
Table 11: Taguchi's Orthogonal Array developed for experimental run 7,8 and 9. 35
Table 12: Aging times selected for solution treated aluminium alloys. 39
Table 13: Images of resulting casting for Al- 4Si- 6Cu- 2Mg- 2Zn- 0.6Mn- 1Sn- 2Ni- 1Ti. 42
Table 14: Deviation of weight for experimental run 7. 42
Table 15: Sample 7 average as- cast hardness. 44
Table 16: Compares sample 7's as- cast and solution treated microstructure. 45
Table 17: Compares sample 7 solution treated and after aging microstructure. 47
Table 18: Images of resulting casting for Al- 6Si- 4Cu- 4Mg- 6Zn- 0.2Mn-.4Cr- 6Ni- 1Ti. 48
Table 19: Deviation of weight for Al- 6Si- 4Cu- 4Mg- 6Zn- 0.2Mn-.4Cr- 6Ni- 1Ti. 48
Table 20: Sample 8 average as- cast hardness. 50
Table 21: Compares sample 8's as- cast and solution treated microstructure. 51
Table 22: Compares sample 8 solution treated and after aging microstructure. 53
Table 23: Images of resulting casting for Al- 4Cu- 6Mg- 4Zn- 0.2Mn- 4Ni- 1Ti. 54
Table 24: Deviation of weight for experimental run 9. 55
Table 25: Sample 9 average as- cast hardness. 56
Table 26: Compares sample 9's as-cast and solution treated microstructure. 57
Table 27: Compares the solution treated and after age hardening microstructure for sample 9. .. 59
Table 28: Revised factor level table with optimum alloying percentage identified. 66
Table 29: Revised orthogonal array table with peak aging hardness and optimum composition highlighted. 67
Table 30: All the solution treatment iteration for sample 7. 79
Table 31: All the solution treatment iteration for sample 8. 80

Table 32: All solution treatment iteration for sample 9..... 80
Table 33: Age hardened sample 7 hardness log. 82
Table 34:Age hardened sample 8 hardness log. 84
Table 35: Age hardened sample 9 hardness log. 86
Table 36: Compilation of data logs for figure 30 to 37 in orthogonal array analysis. 88

Figure 1: FCC crystal lattice of aluminium. [24] 6
Figure 2: Example of orthogonal array. [29]..... 14
Figure 3: Properties of aluminium and alloying elements allowing for high castability. [21]..... 16
Figure 4: Permanent mould casting example. [11]..... 16
Figure 5: Factors affecting selection of casting process. [10] 17
Figure 6: Typical cooling curve of cast. 19
Figure 7: Contact angle in heterogeneous nucleation. [55] 20
Figure 8: Phases of solution treatment. 22
Figure 9: Parameters to be controlled during solution heat treatment. [13] 23
Figure 10: Shows the TTP diagram based on tensile strength. 25
Figure 11: Shows the TTP diagram to obtain ideal grain. [20] 25
Figure 12: Shows the age hardening curve under different aging process. [10] 26
Figure 13: Resulting microstructure of Al-4%Cu. [20]..... 27
Figure 14: How etching creates contrast. [38]..... 30
Figure 15: Schematic of Vickers pyramid hardness test. 31
Figure 16: Possible shape of aging curve. [57] 38
Figure 17: Microstructure of as- cast for Al- 4Si- 6Cu- 2Mg- 2Zn- 0.6Mn- 1Sn- 2Ni- 1Ti..... 43
Figure 18: Sample 7 as- cast microstructure 500x magnification. 44
Figure 19: The plotted aging curve which yields peak hardness for alloy 7 after aging at 200 °C. 46
Figure 20: Porosity at core of sample 8. 48
Figure 21: Microstructure of as- cast for Al- 6Si- 4Cu- 4Mg- 6Zn- 0.2Mn-.4Cr- 6Ni- 1Ti. 49
Figure 22: Shows sample 8 as-cast microstructure at 500x magnification. 49
Figure 23: Solution treated alloy 8 showing fine eutectic phase. 52
Figure 24: The plotted aging curve which yields peak hardness for alloy 8 after aging at 200 °C. 52

Figure 25: Peak aging microstructure of sample 8 at 100x magnification. 54
Figure 26: Microstructure of as- cast for Al- 4Cu- 6Mg- 4Zn- 0.2Mn- 4Ni- 1Ti. 55
Figure 27: Alloy 9 as- cast microstructure at 500x magnification. 56
Figure 28: The plotted aging curve which yields peak hardness for alloy 9 after aging at 160 °C. 58
Figure 29: Porosities seen on sample 9 after heat treatment. 58
Figure 30: Copper influence on hardness of aluminium alloy. 60
Figure 31: Silicon influence on hardness of aluminium alloy..... 61
Figure 32: Magnesium influence on hardness of aluminium alloy. 61
Figure 33:Zinc influence on hardness of aluminium alloy. 62
Figure 34: Manganese influence on hardness of aluminium alloy. 62
Figure 35: Tin influence on hardness of aluminium alloy. 63
Figure 36: Chromium influences on hardness of aluminium alloy. 64
Figure 37: Nickle influence on hardness of aluminium alloy. 64
Figure 38: Full factor table for all experimental run. 81
Figure 39: Full composition of alloys in gram for all experimental runs. 81
Figure 40: All plotted aging curve for sample 7. 83
Figure 41: All plotted aging curve for sample 8. 85
Figure 42: All plotted aging curve for sample 9. 87

1. INTRODUCTIONS

Alloy is a metallic substance consisting of a mixture of two or more elements. One of the elements must be metal. A lot of alloy mixes two or more metal. Alloy is produced by melting down the metal to molten form and then mixing in alloying elements. More importantly are alloy steel, constituting of steels with a significant percentage of elements other than carbon and iron. By alloying, alloy steel can gain various improvement to mechanical properties such as hardness, toughness ductility etc. [1]

Aluminium is one of the most common elements and is the world's most abundant metal as well. Year on year demand for aluminium increases by 29 million tons per year, affirming its importance. Without any alloying, aluminium is soft, ductile, resistant to corrosion, low in density, and conducts electricity. [2] Once alloyed, aluminium will gain superior weight to strength ratio compared to steel with a density of 2.7 g/cm^3, which is one-third the density of steel. [3] The unique combination of light weight and high strength of aluminium alloys allow for the production of strong and light structures and components that are especially essential to the aerospace and aviation industry.

As humanity continues to push the boundaries of technological development, creation of newer and better material such as alloy allows for the applications of said technology. There are millions of alloying combinations yet undiscovered. Each combination may produce a new aluminium alloy with superior, inferior or similar properties to those in the market. The most popular method of discovering these aluminium alloys are by simulation using analytical data software enabling rapid alloy development, revolutionary composition and specific surface technology. [4] Another less used method nowadays is the traditional method of casting and testing random set of alloying composition for specific properties after casting.

Supplemented by design of alloy based upon orthogonal array, experimental process can be optimised to increase the chances of success in finding an alloying composition with superior properties. Orthogonal array works by setting up a series of parameters to obtain different combinations of parameters maximising the combination of parameters for each experimental run. In this case, alloying elements will be the parameter varied to produce each experimental run with a unique combination of alloy. For optimal selection, Taguchi's method orthogonal array allow for the effect of each alloying element to be investigated and studied.

To further amplify the mechanical properties increase after alloying, heat treatment and the aging process will be performed on the alloy sample after casting. Heat treatment is done to alter the properties of steel without affecting the dimensions of the steel. [5] Aging process is used to complement the heat treatment process by subjecting the steel at elevated temperatures for a different rate of artificial aging to manipulate the properties of steel. [5]

The experiment done for the thesis will consist of the hybrid of traditional and modern alloy designing method combining orthogonal array design experiment to cast and test parameters of a random combination of alloying elements with aluminium alloy as the base metal. Aluminium in alloy must consist of at least 63% of the total constituents of aluminium alloy. At the end of each experimental run, a unique aluminium alloy with varied alloying composition will be further divided into multiple samples for individual heat treatment and aging process, further enhancing the mechanical properties of alloy before performing hardness testing and microstructure analysis on each alloy sample.

1.1 Historical Advancement and Motivation of Alloy Development

Historically alloy has been around for over 5000 years. The first alloy developer diluted elemental copper to create bronze with a small amount of tin, ending the Stone Age and initiated the Bronze Age. [43] Humanity has continued alloy development ever since based on this simple discovery where an element possessing attractive properties acting as the master alloy is added on with small amount of other elements to improve the properties. Subsequently, fewer than a dozen of alloy was discovered before the 18^{th} century, options of new development were limited by undiscovered elements which weren't discovered until after the 18^{th} century in quick succession. Of notable discovery, before the 18^{th} century is the discovery of carbon steel by the Persians in the 16^{th} century BC, thus begin the Iron Age. [44] Other alloy developed before the 18^{th} century includes combining gold and silver to create electrum, the formula of 50% gold and silver each is still in use today.

The ancient concept of lightly alloying base metal with other elements has since been increasingly sophisticated. Traditional alloys are developed by trial and error; small amount of elements are added with no resulting product in mind with only an objective to be achieved. This consumes a lot time and is inefficient. Alloy elements are now added in controlled conditions with tenths or hundredths of a percent onto base metal translating to a single alloy having up to a dozen of minor elements. Some current method employs statistical analysis in choosing the constituents or runs

computer simulation to find the best element combination for the chosen base metal. All these alloys will still have a single dominant base element.

Reasons for research and development of new alloy has been mentioned plenty in preceding sections of the thesis report. But studying the historical context of alloy development provides backing on the claims given on importance and reasons for developing new alloys. The claim of new alloy development is integral to the advancement of human technology and advancement can be seen by humanity transition from the stone age to bronze age and then iron age where we arguably still live in with each discovery of new alloy due to the extensive applications of iron and steel in society today. [44]

Aluminium and its alloys are considered recent development as aluminium wasn't discovered until the mid-19th century. Once discovered, aluminium gained immediate prominence and use culminating in the first manned flight by the Wright brothers which utilises aluminium engine. [45]

2. OBJECTIVES

The main objectives of the thesis are:

1. Develop new aluminium alloy with improved mechanical and lower cost compared to currently commercially available aluminium alloy on the market. New aluminium enables more and better option for companies and researcher around the world. With new development, manufacturing process and research can be more efficient and cost can be lower.

2. To understand the effectiveness of orthogonal array for solutes combination. The use of orthogonal array is not common for research and development of aluminium alloy. Through this thesis, the method effectiveness can be monitored for future research and development of new aluminium alloy. Providing an effective and cheaper statistical approach by manually selected alloying constituents as opposed to a programmable statistical modelling using a computer.

3. Analyse the effect of differing combination of solutes on aluminium alloy cast to produce aluminium alloy with high hardness through strengthening by heat treatment. Further

explanation is given in the following sections, but orthogonal array and specifically its variant called Taguchi's Method enable each alloying constituent's effect on alloy properties to be identified. Investigating the effect of alloying elements grants researchers, the ability to fabricate better alloy in future by focusing on beneficial alloying elements by making an educated guess based on scientific result.

4. Getting the hardness of each resulting casted aluminium alloy by performing a Vickers hardness test. Hardness testing gives value to each aluminium alloy for easier comparison based on worldwide accepted testing method and valuation. Provide a means to corroborate with the result seen during microstructure testing.

5. Analyse the microstructure of casted aluminium alloy and after heat treatment. The hardness of alloy is partly controlled by the intermetallic phase of the alloy. Intermetallic phase of an alloy is observable only under an advanced optical microscope. Combining different alloying elements creates different intermetallic phase. Knowing the shape, size, type and composition of each intermetallic phase allow for reasoning and derivation of properties obtained. Comparing as- cast and heat treated sample provide information beneficial or negative response of alloy to heat treatment. Some aluminium alloy does not respond well to heat treatment.

6. Identify the best heat treatment temperature and holding time of each individual alloy casted. Optimum solution treatment and age hardening of each aluminium alloy is different. Certain aluminium alloy may have below average properties at as-cast condition, but correct appropriate heat treatment can exponentially raise the properties of aluminium alloy.

7. Identify potentially functional aluminium alloy for real-world applications. Engineering applications of the results obtained should be investigated to allow for improvement to manufacturing methods and potential technological advancement.

3. LITERATURE REVIEWS

3.1 Aluminium Alloy Metallurgy, Alloy and Alloying Elements

Aluminium alloy can be classified into either wrought alloys or cast alloys. Wrought aluminium alloy is exceptionally ductile and can undergo strengthening processes such as forging, extrusion, rolling and heat treatment. Cast aluminium alloy is aluminium alloy produced in hundreds of compositions by commercial casting process. Poured into the final shape, cast aluminium alloy can only be strengthened by heat treatment.

For the thesis project, aluminium is classified as cast aluminium alloy as it will be produced by conventional gravity casting. Master alloys are alloys added to molten metal to obtain the desired composition and influence the as-cast structure. [22] Composition of master alloy can consist of more than 50% weight of the main alloying element. By melting and mixing constituents one by one into the base alloy, two different type of master alloy can be obtained:

- Concentrated binary master alloys
- Grain- refining master alloys

Binary master alloys are produced by adjusting an alloy composition in small quantities. Composition of alloying elements is equivalent to multiple concentrations of elements in the base alloy. [22] The composition of alloying element added is determined using orthogonal array based on this rule for the thesis research. [23] The goal of casting master alloy is to obtain better grain refinement and desired mechanical properties in base alloy with short alloying time and reduce alloying element losses molten liquid metal. [22]

The disadvantages of casting aluminium alloy are the high shrinkages which cause shrinkages defects, lower ductility and propensity to hot crack.

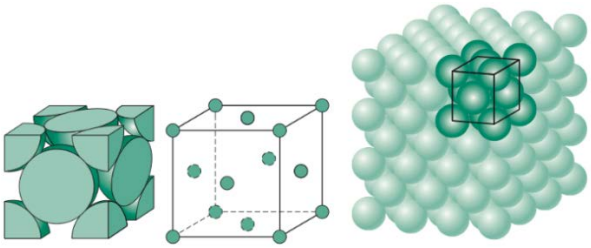


Figure 1: FCC crystal lattice of aluminium. [24]

processing without the risk of failure, but it lacks the strength necessary for most applications due to low hardness and strength. The mechanical properties, primarily ductility of aluminium can be derived from its crystal lattice. The crystal lattice of aluminium alloy is a face- centred cubic lattice. Atoms are present at each corner of the cube with six atoms filling up each face of the cube. The structures pack the atoms as close as possible.

The high ductility, Young's Modulus and softness of aluminium allow for the

3.1.1 Aluminium Alloying Elements, Grain Refiner and Respective Effects

Aluminium by itself is not strong enough for most real-world applications because it has low strength and hardness. A detailed outlook of aluminium base mechanical properties and to be improved upon by adding alloying element is shown:

-Low density	- Excellent corrosion resistance	- High thermal conductivity and melting temperature	- Conducts electricity
- High malleability	- Moderate hardness and strength after alloying	- High ductility	- Reflective

Alloying elements must be added to improve the mechanical and physical properties of aluminium, mainly the strength and hardness. Different alloying elements has its own individual effect on aluminium. Each aluminium alloy produced is given a series depending on its primary alloying element for wrough alloys. The table below details the effect of only the alloying element used for the thesis project and its respective classification if available.

Alloying Elements	Effect and Details
Silica, Si (Major Element) 4xxx series	• Most important and commonly used alloying elements. • Promote high fluidity and low shrinkages to ensure good castability of aluminium. • Reduce the total weight of cast component. • Decreases machinability of aluminium due to the formation of low solubility silica precipitate that in turn, increase abrasion resistance. • Depending on percentage added can form hypoeutectic(<12 wt % Si), eutectic (12-13 wt %Si) and hypereutectic Al-Si alloy(14-25 wt % Si). • Increases solidification time and decreases liquidus temperature. • Silica percentage in aluminium alloy is proportional to the hardness of the alloy, an increase in silica percentage will increase the hardness up to 12% Si and decrease after 15% Si is added. • Up to 17% can be added
Copper, Cu (Major Element) 2xxx series	• Increase strength and hardness at all temperature condition with or without heat treatment from facilitation of age hardening. • Improves machinability by increasing matrix hardness. • Decreases corrosion susceptibility. • Increases stress corrosion resistance. • When heat treated after the addition of copper up to 1.5%, ultimate tensile strength increases due to precipitation of copper-bearing phase in inter-dendritic space. • Usually, only 2% to 10% is added but strengthening properties is maximum between 4% and 6% depending on the influences of other constituents.
Magnesium, Mg (Major Element) 5xxx series	• Increase the strength of solid solution and strain hardening properties; an example is a work hardened alloy. • Increase corrosion resistance and weldability.

	• It is used together with silicon to form a hardening phase of Mg2Si which provide higher strength and hardness. • Prevent the clustering and disperse the graphite particles uniformly because Mg is an active surface agent. • Up to 10% can be added.
Zinc, Zn (Impurities) 7xxx series	• Exponentially increases the strength of aluminium. Result in heat-treatable aluminium alloy with high strength. • Forms precipitate in aluminium matrix. • Decreases resistance to stress corrosion cracking, disallow fusion weld to be done. • Form high-density precipitates. • Up to 8% can be added.
Manganese, Mn (Minor Element) 3xxx series	• Increases the strength through solution strengthening. • Improves low- cycle fatigue resistance. • Negligible effect on corrosion and ductility of aluminium below 0.5% but significant improvement if more than 0.5% is added into aluminium. • Aluminium will not be heat treatable if only Mn is added. • Up to 1.5% can be added.
Tin, Sn (Minor Element)	• Reduces friction in bearing and bushing applications. • Increases conformability and embeddability. • Up to 40% can be added.
Chromium, Cr (Minor Element)	• Enable control of grain structures of aluminium. • It is usually used with other alloying elements, e.g. Al-Mg alloy. • Prevent recrystallisation of alloy during heat treatment. • Reduces stress corrosion. • Up to 0.3% can be added.
Nickel, Ni (Minor Element)	• Improve hardness and strength of aluminium at an elevated temperature up to 600F. • Reduce the coefficient of expansion • Up to 2% can be added.

Titanium, Ti (Minor Element)	• Acts as grain refiner. • Used with boron for better effect because TiB_2 will form. • Always added when application of aluminium requires welding. • Up to 0.35% can be added.

Table 1: Aluminium alloying element effect and details. [26] [27] [28]

The percentages of alloying elements and impurities must be controlled carefully in all aluminium alloy. Wrong balance of constituents adversely affect properties such as strength, toughness, formability, and corrosion resistance. In particular, excessive addition of alloying element into aluminium is not recommended as there is a solubility limit where any additional alloying constituents added will not fully dissolve and mix into the molten aluminium. The undissolved elements will form a different phase and composition possessing different mechanical properties leading to a single cast of alloys consisting of two different metal alloy. For example, at room temperature, copper up to 0.2% can dissolve in aluminium at room temperature but beyond that, the excess copper will form another phase, $CuAl_2$ in addition to the primary phase. [25]

Certain combination of alloying constituents is more compatible and enable more drastic improvement when added together into aluminium. In fact, the combination of MgSi is so widely used it is classified as 6xxx series of aluminium alloy. The combined addition of Mg and Si will enable heat treatability of aluminium alloy. Machinability of aluminium alloy can be improved as well. Wettability of SiC can be increased. Si in matrix provides better elastic modulus, thermal dimensional stability and a lower coefficient of thermal expansion.

For the thesis project, hardness is the primary mechanical properties to be tested with higher hardness desired. To elevate the hardenability of the aluminium alloy casted, alloying elements such as chromium, nickel, manganese etc. can be added. [17] Hardenability is a measure of ease by which a material can be hardened. Each unique alloy with different composition will possess differing rate of hardenability. Although hardenability is usually applied on steel, hardness gained when aluminium undergo heat treatment can be described with hardenability as well. In this case, the hardenability of aluminium alloy is the measure of its ability to precipitate and gain smaller grain upon heat treatment.

3.2 Modern Method to Design New Alloy

In modern times, creating a new alloy is given the term designing an alloy. The difference with the traditional method is the knowledge-guided approach to development and compositionally sensitive design of novel metallurgical material. But having identified beneficial alloys, experiments still need to be done with careful iteration and optimisation. Progressively, more unprecedented alloying elements combination and number of alloying combinations is seen akin to those casted for this thesis itself. To screen and identify entirely new alloy with the old empirical and stepwise trial and error approach is not practical. Systematic method to gain educated guess before casting and experimenting is more probable to succeed instead of blindly casting like the old days. Type of systematic approach available is computational material science includes computational material science, semi-empirical chemical rules based on electronic bonds and structure motifs, combinational metallurgical synthesis. Other methods can be loosely interpreted to consist of identifying the desired property to design and from that initial target design, key parameters are identified to be optimised using associated thermodynamic and kinetic quantities like dislocation properties, certain interface properties, certain phase transformation and more. [58] This thesis intends to adopt the latter method through the means of a variant of design of experiment named Taguchi's Orthogonal Array which will be further expanded upon in the following sections. Key parameters to be quantified in this instance is the alloying elements and respective percentage added.

Significant advantages between development new alloy currently compared to older times are the assess to computers, advanced microscopy along with microanalysis techniques and casting techniques. All which allows for faster and better controls of key parameters quantified to produce better subsequent casting iteration in more efficiently.

A critical aspect of designing new aluminium alloy involves characterisation of structure and chemical composition at nanometre scale using advanced microscopy and microanalysis techniques. According to publication by Ross K.W. Marceau, Thomas Dorin, in modern times advancement in microscopy technique enables for better understanding of materials. [53] Of notable advancement in microscopy technology field includes Transmission Electron Microscopy (TEM) and Atom Probe Tomography (APT). TEM is a rapidly advancing sector of microscopy technology, capable of collecting diffraction patterns from volumes down to few cubic nanometres and procuring image at atomic scale. Spectroscopies done with TEM can analyse the composition

of sample, bonding, optical, electronic structure properties, structure and shape of nanostructure in 3D at atomic resolution if combined with APT according to Giulio Guzzinati et al. [59] For material development, understanding the relation between the structure and composition of microscopic features of new alloy can reveal the origin of properties obtained. TEM is also capable of providing insights to analyse the crystal structure at a specific local scale with higher resolution compared to Scanning Electron Microscope (SEM). APT is the only method that can offer extensive ability to 3D image, measure chemical composition at atomic scale. A key advantage of APT is atomic-scale phenomena such as solute segregation or clustering that effects material properties by control of local phase transformations can be studied. When both APT and TEM are used together, they can achieve concise, accurate and detailed imaging unattainable by other methods.

One recent popular casting advent which is also easily available are 3D printers. 3D printer allows aluminium to be cast to complex shapes through addictive manufacturing method known as Selective Laser Sintering (SLS) or Selective Laser Melting (SLM). Such casting methods may exhibit higher than average properties. In a journal by Francesco Tresvisan et al. they found some final part is able to attain higher strength and ultrafine microstructure from rapid solidification, directional heat flux and temperature gradient. [60] Disadvantages exists however; gas entrapment porosity, oxide layers and material that is not melted are generate frequently. [60] Extensive research is being done to streamline and reduces the defects encountered during such process.

Technological advances in nanoscience make it possible to use nano-size reinforcement in place of micro level. Pre-existing aforementioned modern design method should consider nanoalloy as a viable way of producing new alloys. Nano size reinforcement increases the strength, stiffness, abrasion, wear resistant and reduces density of aluminium matrix composites (AMCs) as quoted from the journal by Aniruddha V. Muley et al. [61] Nanoalloy technology is still relatively new and are only at research stage currently. Nanopowders reinforcement are not cheap as well. Defects such as agglomerations and clustering of nano-reinforcement are common. Nanoalloy is produced by adding nanoscale polymer to the matrix of metal.

3.2.1 Design Of Experiment(DOE)

Design of experiment is a systematic and extensive method to solve engineering-related problems by applying principles and techniques during the data collection stage to ensure the production of a valid, defensible and supportable engineering results. [30] It allows for the experiment to be carried out with minimal expenditure, experimental run and time with control over as much

variable of experiment as possible, resulting in a more efficient experiment. Four engineering problem where DOE can be applied are:

Engineering Problems Area	Descriptions
Comparative	Comparative compares the changes of a single factor on the overall result from improvement or degradation of the process as a whole.
Characterisation	With the identified data, factors are ranked by importance according to the influence of each factor on the process.
Modelling	For this area, the engineer is interested in the functionality of the process with the output being a mathematical function with maximal accuracy to measure the coefficient of the function.
Optimising	Lastly, each process factor is optimised by determining the factor level to have the best possible response.

Table 2: Design of experiments and its area of factor.

3.2.2 Taguchi's Method Orthogonal Array

One type of DOE is the Taguchi Orthogonal Array Designs(OA). The mathematical function employ by this method is general fractional factorial design. It is proposed by Dr Genichi Taguchi based on functional orthogonal design which allows for consideration of selected subset. Advantages of Taguchi's method is its balance which ensures all level of factors are considered on equally whereby each factor can then be evaluated independently of one another. To perform the Taguchi's method, these assumptions have to be made whereby the effects of each factor are separable and has no repercussions on each other. Effect of each factor under these assumptions can be then considered to be linear, quadratic or of higher order with no effect among individual factors. [31] Further advantages are Taguchi's method improves to product quality by emphasizing the mean performance value close to the target value. Taguchi's method is also simple and easy to apply for most engineering cases while allowing for analysis of huge number of parameters without conducting high number of experiments.

Taguchi method further divides the 4 steps of DOE above to a more detailed experimental process, which are:

1	**Selection of independent variables**	Obtaining all valid input for the project/ experiment based on the knowledge of process and outcome desired.
2	**Deciding number of levels**	Number of variable is to be decided. Total number is based on the performance parameter affected by different level settings. If linear performance parameter, the total level shall be two. If there is no linear relationship, the possible total level will be higher than two. Relationship of parameters is largely based on assumptions decided for the project/ experiment.
3	**Selection of orthogonal array**	Minimum number of experiments conducted is to be fixed based on total degree of freedom. Number of experiments to be run has to be more than the total degree of freedom. To calculate the total number of degree of freedom, the experimenter must use one degree of freedom to overall mean of response under study. Number of degree of freedom must be equal to one less than the number of levels available for the factor.
4	**Assigning independent variables to columns**	Independent variables are assigned to each vertical column. A mixed level variable and interaction between variables requires variables to be placed at right columns as stipulated by an orthogonal array. Each variable is to be assigned a level as well.
5	**Performing the experiment**	With orthogonal array established, the experiments are conducted based on the level combination. Interaction of columns and dummy variable column is to be ignored but is required when analysing experiment result.
6	**Analysis of data**	As the experiment is a combination of different factor levels, it is essential to separate the individual effect of the independent

		variable. Corresponding levels for each parameters value is summed up.
7	**Inference**	Higher value of sum of square of an independent variable has more influence on the performance parameter. Calculation of ratio of individual sum of square of a particular independent variable to the total sum of squares of all variables. Ratio calculated will give the percent contribution of independent variable on result allowing for optimal solution to the problem.

Table 3: Steps to conducting Taguchi's orthogonal array.

Test Number	Factors			
	A	B	C	D
1	1	1	1	1
2	1	2	2	3
3	1	3	3	2
4	2	1	2	2
5	2	2	3	1
6	2	3	1	3
7	3	1	3	3
8	3	2	1	2
9	3	3	2	1

Figure 2: Example of orthogonal array. [29]

Orthogonal array is denoted by:

p- the number of rows in array. when combined with k, a single row represent a single experimental run. The total p will the number of test run.

k- is the number of column which also signifies the parameters. This is the total number of variables that that can be handled.

n- Represent the number of entries in the array from 0, 1, 2,… up to n, which is the number of parameters. Also known as the level.

d- is the influence of each entries on array. All orthogonal array has a strength, d. It the multiplications of previous entry of p, n, k multiplied by d. The number of columns required to enable observation of each $Levels^{strength}$ equal.

Applying Taguchi's method allow for the identifications of the important alloying elements to be focused on and composition of alloy be altered accordingly for future research and casting. Effectively eliminating other alloy combination that won't produce casting with desired material

properties. This increase the likelihood of finding an alloy with less experiments and in extension the cost, by approaching the problem with Taguchi's method. [33] For clarification, if a process has eight variables, it would require 3^8 times of experiments which equates to 6561 to properly test all variables. But with applications of Taguchi's method, less experiment needs to be conducted as key parameters can be focused on while parameters with fewer quantitative consequences are effectively ignored. [33]

The main disadvantage of Taguchi's Method are results obtained are relative and does not adequately describe the relationship between parameters and which parameters is exerting the most effect on the final result obtained. [33] For this thesis research, the effect of each parameter can be discovered by complementing the Taguchi's method with Vickers hardness test and microstructure analysis. By performing both analyses, the result obtained can be cross-referenced with the alloy composition of the orthogonal array and major elements of each experimental run. Effectively compensating for the major disadvantage of Taguchi's method.

3.3 Casting Metal

Casting is a net shape manufacturing process where molten metal/steel/alloy is poured into prepared cavity of desired shape and leaving it to solidify into the desired shape. In a singular step, metal can be cast into simple or complex shapes for applications such as manufacturing of car components, casting of intricate shaped sculpture or mechanical properties testing such as for this thesis project. Properties variation in alloy is possible by varying the composition of the elements during casting. Testing of mechanical properties of cast alloy is popular as testing can be done via non- destructive testing.

From previous literature review discussing the methods to develop new aluminium alloy and orthogonal array, it is implied to conduct this thesis research, aluminium alloy is to be casted as none of the newer and more advanced methods is to be employed. Several samples are will be cast for a trial and error based experiment and as such suitability and merits of casting aluminium alloy is shown in figure below.

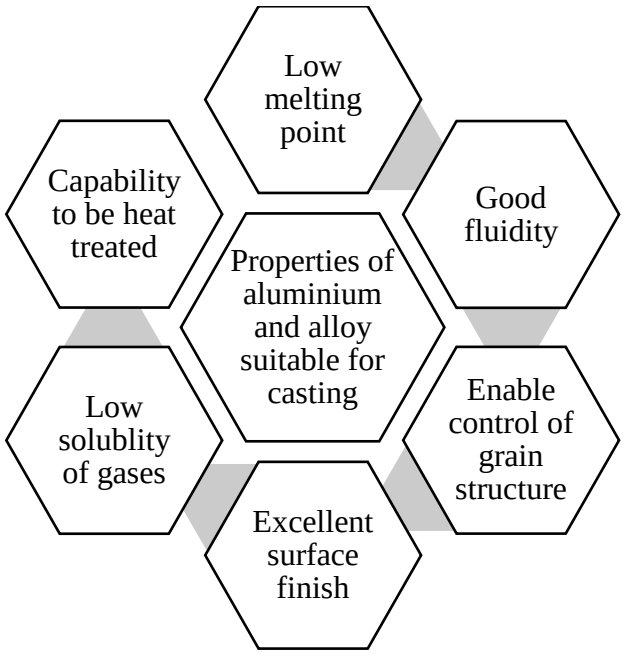


Figure 3: Properties of aluminium and alloying elements allowing for high castability. [21]

3.3.1 Permanent Mould Casting and Justification

There are various casting methods such as sand casting, plaster cast, investment cast etc. differentiated either by mould used or casting process. Casting is generalised into two expendable mould types or multiple- use mould type. The casting process used for the thesis project is permanent and semi-permanent mould casting which is a multiple-use mould type. Semi-permanent mould is the typical casting process for the production of aluminium alloy using re-useable metal moulds. Base metal is melted in a furnace above melting temperature to molten state before alloying elements is slowly added in.

Once the metal and alloying elements have fully melted and mixed, molten alloy is poured out to fill out the cavity of metal mould. [7] The metal mould is re- usable again.

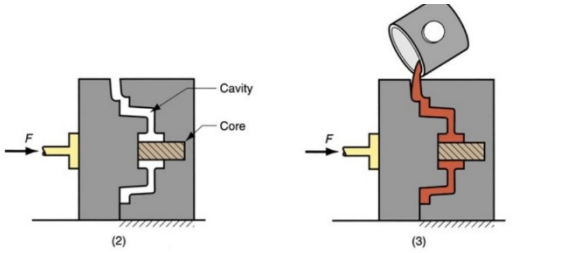


Figure 11.7 - Steps in permanent-mold casting: (2) cores (if used) are inserted and mold is closed; and (3) molten metal is poured into the mold. As the metal solidifies, the mold is opened and the part retrieved.

Figure 4: Permanent mould casting example. [11]

Semi-permanent and Permanent Mould Casting	
Advantages [11]	**Disadvantages [12]**
• Exceptional mechanical properties which are better than sand casting, especially in the strength. • Excellent surface finish provides a dense surface structure which is easier to polish. • More controls over parameters of casting to ensure accurate results. • When compared to sand casting, high weight reduction in casting can be achieved. • Fewer part variations to ensure almost uniform part is obtained for each casting. • Efficient repeatability for casting.	• High mould cost. • Hard to cast high melting point metal into metallic mould as metallic mould may melt. • Can't cast shape which is intrinsic and complex. • Can't cast large parts.

Table 4: Details of semi-permanent and permanent mould casting.

Each casting process has its advantages and disadvantages; selection of appropriate casting process is dependent on:

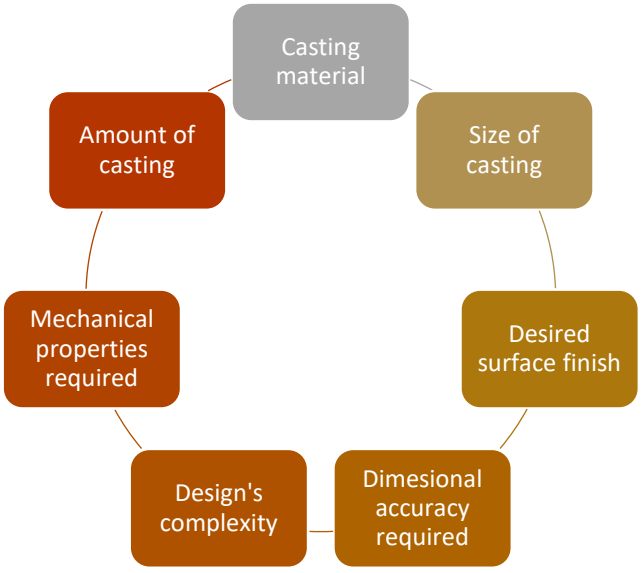


Figure 5: Factors affecting selection of casting process. [10]

The selection factors that are important for the success of the thesis project is identified as amount of casting, mechanical properties and casting material. Thus, permanent mould casting is selected for casting of thesis project primary primarily for the ability to cast repeatably in an efficient

manner as the same mould can be used for casting multiple times. If the mould is reusable, a lot of time and effort can be reduced as the thesis project requires casting of multiple alloying samples. Parameters of the casting can be better controlled and kept constant to ensure more accurate results can be obtain. The main purpose of the thesis project is to develop a new aluminium alloy with superior properties. Using permanent mould casting method increases the chances of obtaining better mechanical properties on the casted alloy because permanent mould casting produces casting with greater mechanical properties. The low weight of the aluminium alloy which is a unique physical property of aluminium alloy with good strength to weight ratio can be reduced as well by using permanent mould casting. Disadvantages of permanent mould casting are not important because the thesis project does not require the casting of high melting point metal, intricately shaped part or large parts.

3.3.2 Aluminium Casting Fluidity

Regardless of casting method employed and metal to be cast, casting fluidity is a key aspect in prevention of flashing, shrinkages and porosity when casting all metal. Fluidity is the ability of molten metal to fill narrow spaces. Low metal fluidity causes shrinkages and porosity while high metal fluidity makes flashing at mould joints and surface roughness. Getting the right level of fluidity will produce an as-cast metal which almost no or no defects.

Pure aluminium has considerable degree of fluidity even if it is cast at just above the liquidus temperature. The fluidity of aluminium decreases as purity of aluminium decreases. Exponential decrease in fluidity is proven even with only 1% decrease in purity. Aluminium with 98.6% purity only has 70% of fluidity possessed by a 99.8% pure aluminium as stated by journal by Francoise R. Mollard etc. [56]

Addition of alloying elements in most cases decreases casting fluidity. Among the scientific community and engineers, it is widely accepted that alloying elements contribute to the breakdown of smooth solidification front that exists between solid and liquid in pure metals. This produces a “mushy” freezing alloy that stops flow earlier than that of pure aluminium. Of course, other factors such as amount added and proximity to eutectic effect the fluidity according to journals. [56] A common practice is to add titanium and/or boron as grain refiner during casting. Fluidity decreases in inverse to higher amount of grain refiner. From the same journal by Francoise R. Mollard etc, current technology combines titanium and boron in Al-5Ti-1B matrix; usage allows for grain refining properties to be achieve even with less than 1% addition. [56] Another influence on

fluidity is the type of mould used along with the mould coating used. Selection of mould used is dependent on type of casting desired and applications of casting so there are not much control available. However, selection of appropriate mould coating can help reprimand these effect. Thus, selecting an appropriate mould coating is important and well documented. Mould coating reduces the heat transfer rate between mould and flowing metal. Coating acts as insulation and form heat transfer barrier which allow for better mould filling which as mentioned fluidity is a measure of liquid metal ability to fill the mould.

3.3.3 Cooling and Solidifications

Cooling and solidifications are two important properties governing the resulting casting and the quality of the casting. Solidification is a process where liquid converts to solid and is done by the production of crystalline through nucleation and growth of grains. [9]

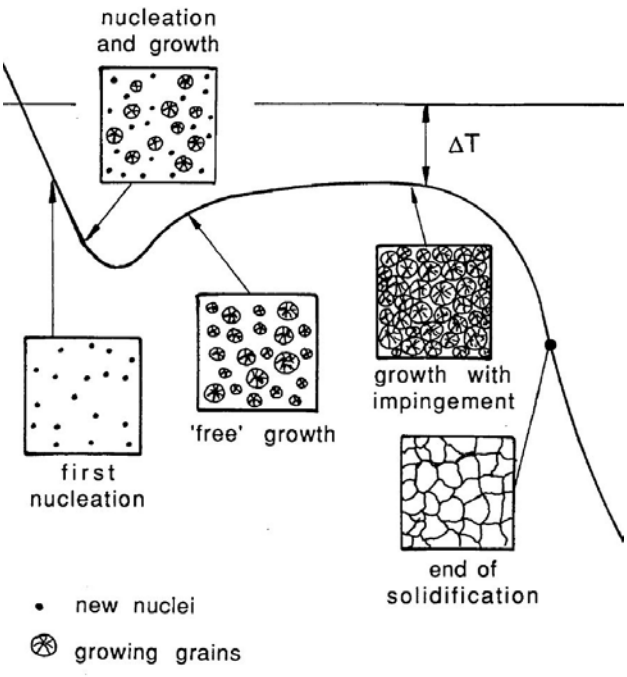


Figure 6: Typical cooling curve of cast.

Solidification of molten metal can be described in figure 6. First molten metal is cooled a few degrees to the liquidus temperature where nucleation of grains will occur. Next, the molten liquid is supercooled to beyond the liquidus temperature momentarily for constitutional undercooling. It is the zone ahead of the interface due to the rejection of solute by solid. Constitutional undercooling will induce the growth of nucleation. Nucleus will grow in the direction opposite of heat flow to form dendrites that is either columnar or equiaxed as long as the constitutional undercooling zone is big enough. Free growth will continue as temperature drops as the dendrites grow freely in mushy zone consisting of solid and liquid until the branches of dendrites come in contact and are packed with each other, grains will then form and crystallisation will end the dendrites combines. Once crystallisation ends, the temperature will drop rapidly and molten metal will completely solidify.

Nucleation can be categorised into homogeneous nucleation and heterogeneous nucleation. Theoretically, there is only one temperature where pure solid metal can co-exist with pure liquid metal. The free energies of solid and liquid are equal at this temperature in correspondence to the equilibrium melting temperature, T_m also known as temperature of melting of solid. At temperature lower than T_m, solid will be in a stable state and have lower free energy. Metal tends to solidify when its temperature is below T_m due to decrease in free energy during solid- to- liquid transformation. Primary driving force of homogeneous nucleation is the degree of undercooling, both increase in proportion with each other. To achieve homogeneous nucleation, there must be superheating, undercooling and closed packed atomic clusters of significant size to prevent preferential cooling. These conditions are hard to realise. Generally, heterogeneous nucleation is more likely to occur. Contrary to homogeneous nucleation, site of nucleation for heterogeneous nucleation initialise from solid surfaces in liquid state. Solid surfaces are a broad term which covers surfaces of foreign substances, mould or insoluble impurities which are preferential site for solidification. Solidification by foreign substances is governed by the wetting angle. The driving force for heterogeneous nucleation is the angle of contact, θ, between the substrate and solid metal. [55]

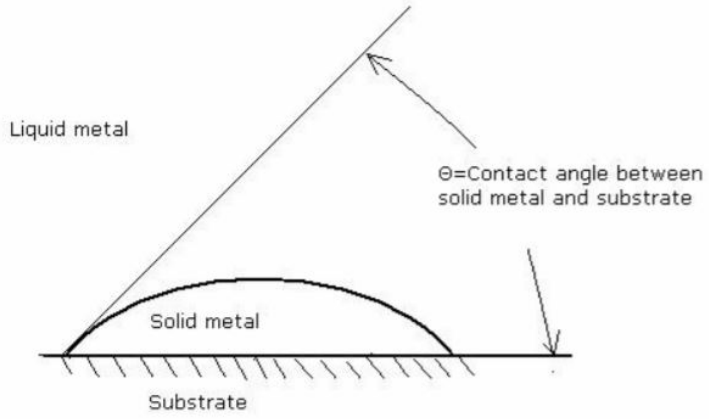


Figure 7: Contact angle in heterogeneous nucleation. [55]

Nucleation on these sites is easier when the angle of contact, θ is small causing lower interfacial energy, stimulating the formation of solid- metal nucleus on the surface of substrate. Angle of contact, θ is maximum at 180^o, where solid/substrate interfacial energy is high and nucleation is

similar to homogeneous nucleation. Contact angle is controlled by lattice spacing and chemical nature of substrate surface. [55]

3.4 Heat Treatment

Aluminium alloy is an alloy which strength primarily originates from elements in solid solution and grain size. [12] In the purest form, aluminium does not respond well to heat treatment. Alloying with metallic element will enable thermally transforming capabilities to become heat treatable. Heat treatable alloy usually contains one or more soluble alloying constituent such as zinc, copper, lithium, magnesium and silicon. Added individually or with other elements, secondary phases that strengthen the alloy will form by improving the solid solution, grain size and allow for precipitation of fine intermetallic compound. All which is central to the thesis research. [46] As mentioned in section 3.1, all alloying elements have limited solubility in aluminium, causing aluminium alloy to have impurities such as silicon or iron. Impurities in aluminium combine to form morphology of phases that cannot be affected by heat treatment. Regular heat treatment of such alloy will not produce any improvement to mechanical properties and in some instances, serve to deteriorate the mechanical properties of aluminium alloy. Aluminium alloy undergoes different and unique heat treatment compared to conventional heat treatment consisting of a three steps process in order of:

1) Solution heat treatment at temperature above solvus.
2) Quenching to induce quick solidification.
3) Age hardening by heating the alloy to a certain temperature and holding it for a specific period of time.

3.4.1 Solution Heat Treatment

According to a journal published by Emma Sjölander and Salem Seifeddine, ‘The heat treatment of Al–Si–Cu–Mg casting alloys’, the purpose of solution heat treatment is to: [47]

- Dissolve as many soluble phases formed during solidification as possible, examples are phases containing copper, magnesium etc.
- Homogenising all the alloying elements into a lesser number of phases.
- Spherodise eutectic particles formed from alloying elements.

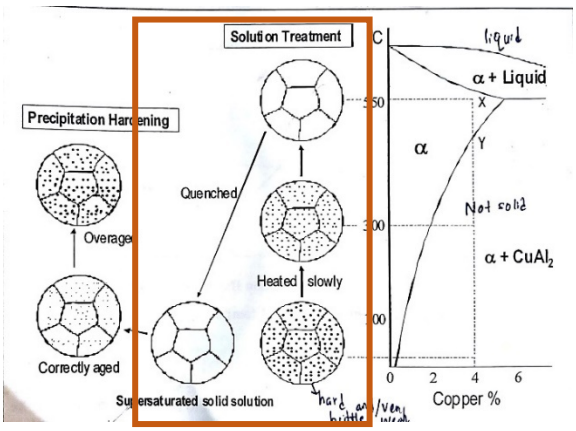


Figure 8: Phases of solution treatment.

Example of a conventional solution heat treatment process of aluminium alloy equilibrium diagram with one alloying element is described based on figure 4. Normally when cooled from molten inducing temperature to 550 °C, crystal of alpha solid solution of the alloying element will form in aluminium. Solid solution will slowly become saturated as it drops towards Y temperature. As cooling continues after temperature Y, saturated solid solution will transform into precipitate. Once room temperature is reached, structure of alloy will consist of alpha phase solid solution containing a coarse precipitate of alloying element in aluminium intermetallic compound. In this phase, the aluminium alloy is softer, ductile and weaker. However, if alloy is quenched instead of naturally cooling from X temperature to induce quick cooling of alloy, equilibrium conditions can't be achieved due to shorter period of cooling time. Equilibrium conditions can only be achieved when time is long enough to allow for grouping together of atoms through diffusion which forms precipitates. As such quenching will prevent the formation of precipitation, resulting grain will consist of supersaturated solid solution of alpha phase alloy at room conditions. Fundamentally the previously described process is known as solid solution treatment and is a mean for aluminium alloy to softened and prepared for cold working. Grain formed from alpha phase solid solution will be finer and will age to form some precipitation over time. The optimal amount of precipitation promotes higher hardness and strength than when cooling under equilibrium conditions yet still being able to retain the original ductility.

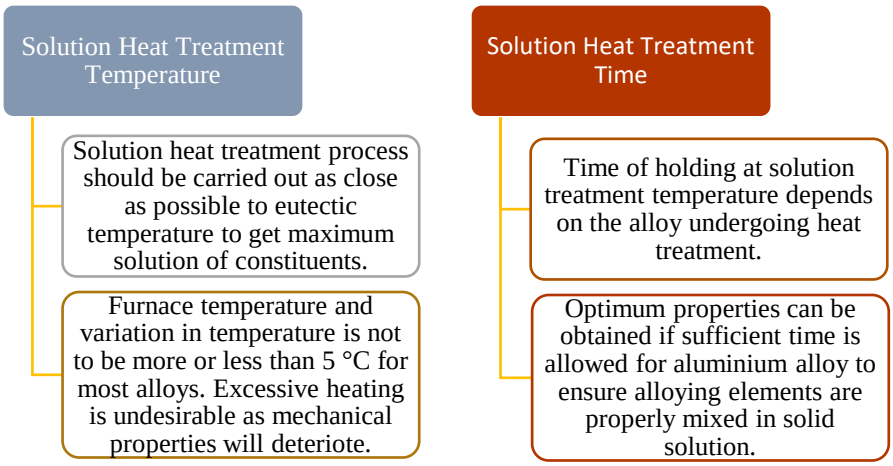


Figure 9: Parameters to be controlled during solution heat treatment. [13]

The journal by Emma Sjölander and Salem Seifeddine notes the limitation to solution heat treatment of aluminium alloy containing Si- Mg- Cu constituents and its concentration. Maximum solution heat treatment temperature with copper and magnesium is prone to incipient melting arising from segregated phases composed of solidified melt that is rich in solute elements The localised melting severely compromise the mechanical properties of the alloy. [47] The recommended temperature from further reading revealed that for alloys containing copper, solution treatment temperature must be lower than 540 °C. [48] With lower Mg concentration, the solution heat treatment temperature will cause partial melting starting from temperature above 519 °C. [49] To get an accurate temperature range is heavily dependent on casting solidification rate and heating rate of solution treatment.

It should be noted from purpose of solution treatment is to dissolve as much phase as possible as not all phases can be dissolved, this phenomenon is known as dissolution. Different particle from combined constituents has different solubility such as θ-Al2Cu is easier to dissolve while particle such as Q-Al5Cu2Mg8Si6 will be harder to dissolve. A perfect solution treatment will be for all phases form a homogeneous solid solution. Homogenisation entails detachment of atoms from coarse particles formed during solidification, leading to diffusion through matrix to decrease the concentration gradient. [47] This is related to the solution treatment time as solution treatment time is determined by the nature of the diffusing atoms.

3.4.2 Quenching

Quenching is part of solution heat treatment, done to ensure that dissolved alloying elements remain embedded in solution when cooled to room temperature, inducing a hardening effect on alloy. [13] There are various type of quenching media enabling different rate of cooling on the alloy:

- Caustic solution
- Brine (10% salt solution)
- Water
- Polymer
- Oil
- Molten salt
- Air

Among these quenching media in figure 9, water, brine, air and oil is the most commonly used. Air quenching is usually for low alloy steel, irrelevant to the thesis project is thus not detailed in table below:

Quenching Media	Description
Water	Water is cheap, easily available and stored. It provides a fairly fast quenching. A problem of water quench is the formation of soft spots due to bubbles.
Brine	Brine provide the fasters quenching but also has the most drastic effect on quenched alloy. Cracks and distortion can easily occur if the alloy quenched do not has good hardenability.
Oil	Oil has the slowest cooling rate and the least severe quenching consequences such as cracks and distortion. Slower cooling rate leads to a milder temperature gradient.

Table 5: Shows commonly used quenching media and description. [18]

Choice of quenching media used is dependant on the critical cooling rate(C.C.R) of alloy hardened. Slower cooling rate will induce harder structure where cooling rate is to exceed the C.C.R. Quenching of aluminium alloy does not produce martensitic microstructure that occurs for regular steels. For most alloys, precipitation begins at temperature below 450 °C; alloy must not be below said temperature as it renders quenching process moot.

Theoretically, as for the thesis project, quenching should be done as fast as possible to suppress decomposition of solid solution, but consideration should be given on the residual stress and

distortion from such fast cooling. Resulting mechanical behaviour after heat treatment can be predicted by plotting cooling curve on the Time- Temperature- Precipitation(TTP) diagram which is analogous in principles to Time- Temperature- Transformation(TTT) diagram. [14]

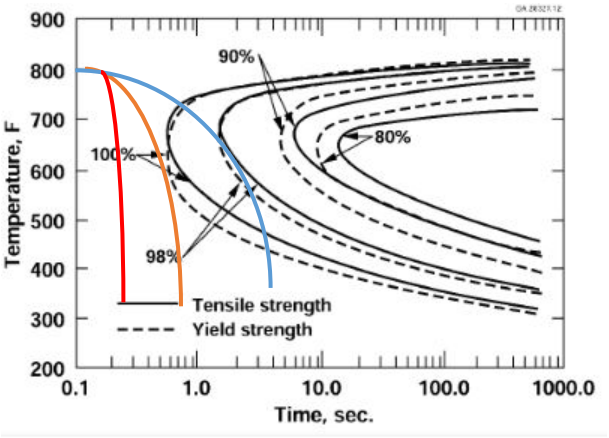


Figure 10: Shows the TTP diagram based on tensile strength.

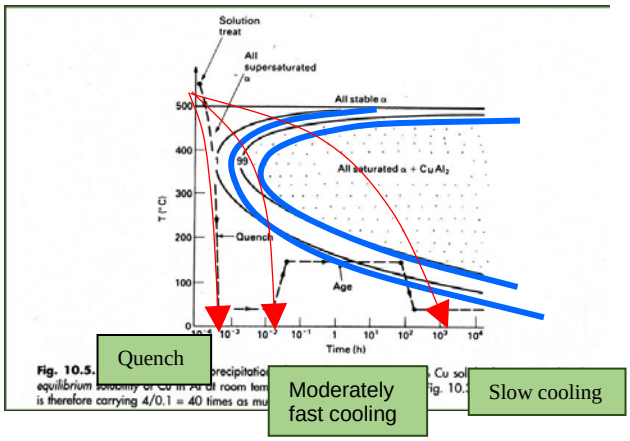


Figure 11: Shows the TTP diagram to obtain ideal grain. [20]

The statement of faster cooling rate form quenching is more favourable is proven in the TTP diagram in figure 7. On the figure is an orange and blue plot which signifies cooling curve of aluminium alloy. As seen on the slower 'blue' cooling rate, the resulting strength of the aluminium alloy is reduced. The 'orange' curve possesses a faster cooling rate and just misses the nose of TTP curve and is able to produce an alloy with 100% mechanical strength. This cooling curve is the aforementioned critical cooling rate and is important for the production of alloy with better mechanical properties which is one of the objectives of the thesis project. [15] Cooling rate is not to be too rapid and severe, 'red' curve on figure 7, as compared to the critical cooling rate of the alloy due to cracking and distortion. One way of controlling the cooling rate is by choosing the appropriate quenching media. If multiple quenchants can produce desired properties on alloy, quenchant with lower cooling rate should be used to produce a less drastic effect.

3.4.3 Age Hardening

Supersaturated solid solution of alpha phase formed from controlled quenching is not stable at room temperature and tends to precipitate over time. Fine particles of evenly distributed precipitate will form, giving the alloy enhanced strength and hardness compared to coarse precipitation formed through equilibrium conditions cooling. This statement is validated by J.F Nie and BC. Muddle which studied how different precipitation strengthens the aluminium alloy. In their research, they conclude high strength light alloy is strengthened by precipitation or dispersion hardening. Form and shape of precipitates include uniform, fine-scale dispersion of plate-, lath- or rod-shaped

precipitates of high aspect ratio. [39] From the same journal it states that to achieve highest yield strength, aluminium alloy is to be strengthened by thin plate-shaped precipitates forming parallel to the primary slip planes of α-Al solid solution (i.e. {111}α). [39] A optimum age hardening process will have clusters of fine precipitate named Guinier–Preston zone(GP zone). GP zones with clusters of solute atoms that are coherent to the lattice of aluminium alloy enabling aluminium alloy to achieve higher mechanical properties. [42]

Control of age hardening can be done by natural aging or artificial aging. Natural aging occurs naturally over a period of a few days. While artificial aging causes precipitation to happens faster by reheating the alloy to about 115 °C to 200 °C for a period of within minutes of artificial age hardening up to a few days. [40]

Selection of aging process is determined by the speed of precipitation of alloy and tendency of alloy to form the right amount of precipitation over aging period. Alloys which can't form sufficient precipitate over natural aging cycle is always artificially aged. In some cases, natural aging can be suppressed by subjecting the alloy to temperature at or below 20 °C. Suppressing the onset of aging on alloy is to allow for forming process to be done on the alloy. Once alloy age, hardness of alloy will increase proportionally, making work forming impossible on the alloy without causing cracks and distortion to alloy.

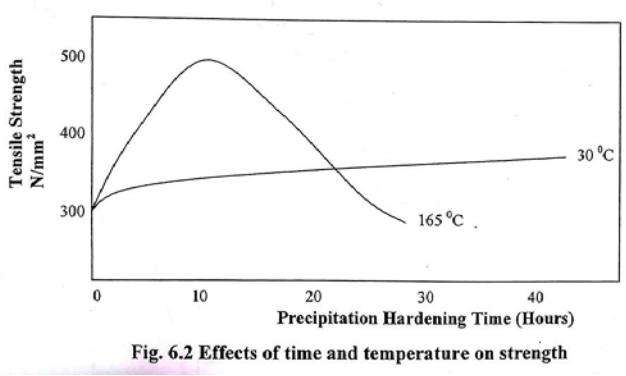


Figure 12: Shows the age hardening curve under different aging process. [10]

Figure 12 shows an example of the effect of aging time and temperature on the strength of the steel. It can be inferred from figure 12 that maximum tensile strength can be achieved through artificial aging. As alloy ages, fine precipitates of alloying elements increasingly form, promoting the strength and hardness of alloy. Dislocation movement is impeded by these fine precipitates, forcing the dislocation movement to cut through the hard precipitate or go around the grains of the precipitates. Prolonged treatment beyond maximum strength is not recommended however as

strength of material will decline after maximum strength is achieved as the precipitation of coarse precipitate. This phenomenon is known as overaging. [13]

Choice of temperature should not be too high as high temperature and long aging time will result in lower yield strength, ultimate tensile strength, and fracture elongation according to research published by J.-P.Bourgeta, M.Fafardb, H.R.Shakeric and T.Côtéd. [41] The same journal states that increasing the temperature allows for peak hardness and elongation to be reached faster but the hardness obtained is also significantly lower. [41] Other studies by Siddiqui et al. report that increasing aging time is inversely proportional to elongation of the aluminium alloy. [71]

3.5 Microstructure of Aluminium Alloy and Microstructure Analysis

3.5.1 Microstructure of Aluminium Alloy

Microstructure of aluminium alloy starts forming when it turns into solid solutions. Generally, microstructure of aluminium consists of saturated alpha matrix of aluminium and precipitates of alloying elements+ aluminium which has clustered together. Size and dispersion of the precipitates are determined by the cooling rate which the aluminium alloy is subjected to.

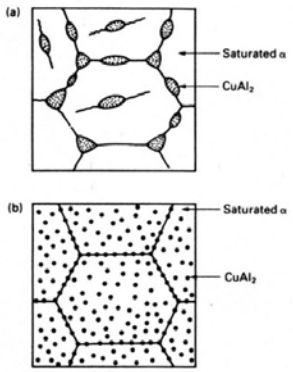


Figure 13: Resulting microstructure of Al-4%Cu. [20]

Slower cooling will lead to production of large and far apart precipitate that is deposited near the grain boundaries of the grains. A faster cooling rate will produce finer, closer and well disperse precipitate. These fine precipitates is deposited all over the saturated alpha phase grain. With even faster cooling rate at critical cooling temperature, supersaturated solid solution of alpha phase can form where it is a state where most material has are able to dissolve into the matrix. Supersaturated solid solution is desirable as all the alloying elements added to the solvent has fully dissolve clustering into maximal amount of small and fine grain upon solidifications. [20]

The ideal microstructure will of course be the one that has large deposit of fine grains around the microstructure of aluminium alloy prevent dislocation movement.

3.5.2 Microstructure Analysis

The purpose of microstructure analysis is to evaluate the microstructure of an alloy at the magnification, which provides the most clarity. Microstructure analysis can evaluate the presence of defects in alloy, type and size of grain and its grain boundaries. Steps and details of microstructure analysis are detailed in the table below.

Steps	1) Sectioning	2) Mounting	3) Grinding	4) Polishing	5) Etching

Table 6: Preparatory steps for microstructure analysis. [19]

All cutting process will introduce damage to the alloy, but proper method of cutting can minimise the damage. Abrasive cut- off saw can produce smooth surface with minimal damage. Parameters such as the thickness of wheel, proper coolant, feed speed and more need to be controlled to have optimal cutting conditions. Dry cutting without coolant is not advisable. Cutting with abrasives will cause a large amount of damage as well, the diamond saw will produce less damage. [38]

The second step is to mount the specimen for easier handling whilst minimising the damage on the specimen itself. Generally there is no limitation to mounting material to be used on specimen whereby the general rule is only that the mounting material adheres well to the specimen. But mounting material must be electrically conducting if the specimen is to be electropolished or examined under Scanning Electron Microscope to understand the constituents of each microstructure features. Mounting is classified into hot mounting at about 200 °C mounting press or cold mounted using thermosetting resin and thermosoftening plastic. Cold mounting is only used if there is large amount of specimens or hot mounting will negatively effect the specimen. Material for hot mounting includes polyfast, multifast etc. If material is porous, resin is used to impregnate the mounting to prevent contaminants and etchant being trapped in pores.

Grinding the surface layers damaged by previous steps is the third step. For easier grinding, mounted specimens is grinded automatically on machine with rotating disk and abrasive paper flushed with suitable coolant to remove debris and heat from grinding. Specimen is grinded from coarser paper to smoother paper progressively with the coarseness indicated by number where lower number paper coarser than higher number paper as it is determined by number of silicon carbide per square inch. Each follow up grinding will remove scratches of the previous grinding and can be done easier by orienting the specimen perpendicular to the scratches. Following a grind, specimen is to be washed before continuing to the next roughness grade. There are many different

grade of roughness, and first grinding paper to be used can be determined by the equation below because starting from the roughest grinding paper is not efficient and cost effective.

$$paper\ roughness = \frac{16000}{width\ of\ largest\ scratch(microns)}$$

For the fourth steps, the grinding paper is replaced with polishing soft cloth which is impregnated with diamond particles and oily lubricants. Likewise, the specimen is to be polish from rougher grade to smoother grade of polish. After each polish, the specimen surface is to washed thoroughly with alcohol and water to prevent contaminations. A layer of undisturbed material on surface of specimen usually arise from mechanical polishing especially if excessive force is used to grind or polish. To remove the mechanical damage such as plastic deformation below the surface, electropolishing or chemical polishing can be employed

Quoted from 'Journal of Material Processing Technology' etching is mentioned as corrosion damages materials under normal circumstances, but it can be applied as an efficient method for shaping materials that is named "etching". [37] Features that will be reveal includes shape and size of grains, phases, porosity, intermetallic, segregation and inclusion which is present on surface of alloy. Etching is not always required if features are already visible after polishing. [36]

There are 3 types of etching which are chemical etching, electrolytic etching and heat tinting. Chemical etching immerses the surface of metal with etchant and selectively corrodes the surface of metal. Time of etching and type of etchant is to be properly controlled depending on the type of metal. Electrolytic etching uses low current and voltage density. Sample acting as anode and highly insoluble conductive metal will act as cathode and complete an electrolytic cycle to etch the sample. Both the sample and cathodic metal must have different electromotive force of the metals. Heat tinting oxidises the sample in furnace inducing surface features at different rates to reveal said features. [36]

For aluminium and aluminium alloy, chemical etching is the preferred etching process and has been industrially completed by M.C. Sanz. [37] Following that, various industrial reagents was developed such as Keller's reagent, Graff- Sargent reagent etc. It should be noted that various new chemical solutions are being tested to produce a more efficient and desirable etching such as a recently published journal studies the effect of ferric chloride (FeCl3). Ferric chloride is a cheap

and easy to control etchant and has been applied industrially for steel and copper alloys. The journal concludes by saying ferric chloride is a very suitable etchant for aluminium alloy chemical reaction is simple and process easy to control, applicable for situation where weight reduction of material is important. [37]

The most important parameter of chemical etching is the selection of etching and the following etching time. An effective etchant can produce a smoother surface finish coupled with a higher etch rate. [37] Etching can also remove a thin and deformed layer introduced by grinding and polishing the metal. Rate of etching is affected by the crystallographic orientation, the phase of alloy and region of stability. Etchant will attack high energy sites such as boundary and defects to create contrast between features. [38]

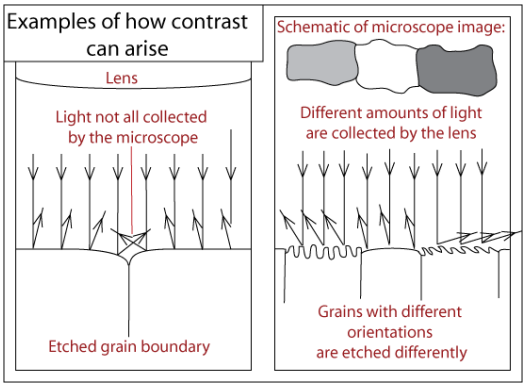


Figure 14: How etching creates contrast. [38]

After etching, various pits may be introduced to the surface of metal. These are result of localised chemical attack which should not be treated as features of the metal. Care should also be taken to prevent from over-etching of samples. Etching for prolong period will promotes growth of pits, obscuring the desirable features to be observed. When over-etching occurs, surface of metal needs to be grinded and polished again. Surface to be analyse must also be flat to obtain an clear view on the microstructure as surface which is not flat may bend the light from the microscope creating a distorted view.

3.6 Hardness and Hardness Test

Hardness is defined as the ability of a material to resist indentation, abrasion and/or deformation. The reason TTP diagram which measures the tensile strength of aluminium alloy is relevant, although the main properties to be enhanced is hardness, is because hardness and tensile strength is proportional to each other. An increase in tensile strength will increase the hardness of aluminium

alloy as well. Level of strength of material and resistance to wear can then be tested using hardness test as well. Ductility on the other hand is inversely proportional to hardness, increase in hardness will decrease the ductility of material.

One mechanism in relation to hardness is the dislocation movement. Dislocation movement can be classified as non-parallel and parallel plane dislocation movement. For parallel plane dislocation movement, movement will be easier due single line of movement, resulting in vancancies or interstitial spaces. Dislocation movement on non-parallel plane is harder as the planes will hinder each other by producing sharp break. There are few methods to suppress dislocations movement which are adding foreign elements, substitutional atoms, grain boundaries, external grain surface and phase change. [72] This ties to the reason why heat treatment of aluminium alloy allows for higher hardness as phase of alloy is changed and grain boundaries forms from nucleation of small intermetallic particles and why selecting the right alloy constituents is important as it determines the formation of above mentiond factors governing dislocation movement.

Three regularly used hardness test method:

1) Brinell hardness test 2) Vickers pyramid hardness test 3) Rockwell test

For the aluminium thesis project, the focus will be on the Vickers pyramid hardness test. Vickers pyramid hardness test forces a diamond indenter into test material under a constant load. Geometry and dimensions of diamond indenter is shaped like a pyramid with a 136^{o} apex angel between opposite face of indenter. [10] Load applied should be as high as allowable to prevent the errors arising from variation between surface layer and bulk of material. Percentage error from reading the diagonal will also be reduced as larger area of indentation will be easier to observe.

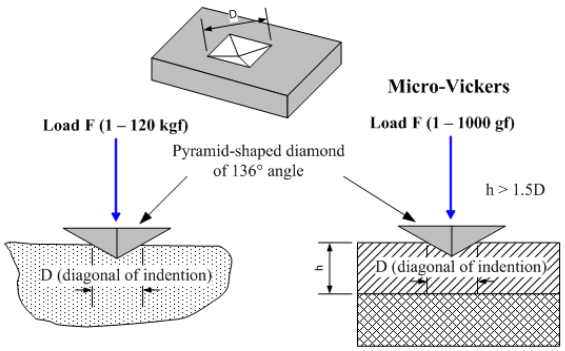


Figure 15: Schematic of Vickers pyramid hardness test.

Value of material hardness can be calculated by measuring the resulting D, diagonal of indentation across all 4 sides to get the area after the load is removed by applying:

$$H.V = \frac{Load\ F(kgf)}{Area\ of\ diagonal\ of\ Indentations} = \frac{F}{D^2/1.854} = 1.854\frac{F}{D^2}$$

Advantages	Disadvantages
• Impression made are similar and consistent geometrically, result obtained is more accurate because there is no variation to depth of impression. • Hardness result for material possessing high are more accurate. • Size of impression is small and produces negligible indentation to workpiece tested. • The same indenter can be used for both soft and hard material and need not be changed as compared to Rockwell hardness test. • Tester do not need to select a F/D^2 ratio such as Brinell test.	• More surface preparation of material is needed, polishing and grinding. • Indenter cost is expensive.

Table 7: Details the advantages and disadvantages of Vickers pyramid hardness test. [10]

Applications of Vickers pyramid hardness test is more suitable for the aluminium thesis project because of ease of testing. The process of using Vickers pyramid hardness test is easier and simpler compared to other hardness test method. For example Brinell hardness test which requires the selection of F/D^2.While more surface preparation such as polishing and grinding is required to perform Vickers pyramid hardness test, it won't be a fuss as these surface preparation process will be done either way to conduct microstructure analysis. Better result accuracy can be obtained as well by applying Vickers pyramid hardness test.

4. METHODOLOGY

4.1 Material and Preparation

For this thesis experiment, aluminium alloy was used as the master alloy along with aluminium alloying elements of silica, copper, magnesium, zinc, manganese, tin, chromium, nickel and titanium which was researched. All metal used was as cast from the manufacturer coming in blocks. Based on the literature research done beforehand, the alloying percentage of each element was determined to be between:

Si%	Cu%	Mg%	Zn%	Mn%	Sn%	Cr%	Ni%	Ti%
3- 8	2- 6	3- 8	2- 8	0.1- 0.6	< 4	0.1- 0.6	0< 3	1

Table 8: Allowable percentage of alloying to be added for to aluminium alloy.

The specific percentage to be alloyed for each element is to be determined with the factor level assigned for each element of orthogonal array. Once the required alloy is determined, the availability was checked for in the foundry. It was found that there was no pure silica, chromium, nickel, and manganese available so Al-50%Si blocks, Al-60Mn, Al-10Cr(g) and Al-19Ni(g) was used instead.

Originally boron was supposed to be added into the matrix as well, but there was a no boron available in the steel foundry. Absence of Boron is not important regardless because the primary purpose of adding boron on aluminium alloy is for grain refinement which can be achieved by adding titanium in small percentage as well.

Hardness of the aluminium master alloy was not noted. But from manufacturer note, it can be assumed to be over 99% pure with a 30 HV hardness. [32]

4.2 Constructing Orthogonal Array

Having identified the composition of each metal to be used. Orthogonal array can now be created basing on figure 3 shown in literature review. The test number in this case will be the aluminium alloy to be casted and the factors level being the percentage of alloying elements added. Each test run will produce an aluminium alloy with differing composition as a result.

An orthogonal array of size 8x16 was chosen, meaning there is 8 columns of variables to be controlled, ‘k’, where alloying elements is assigned to. The 8 alloying elements are magnesium, silica, nickel, tin, manganese, zinc, chromium and copper. The ‘p’, 16 experimental runs are

divided among 5 thesis students. Experimental run focused upon by this thesis is experimental run 7, 8 and 9. Factor level,'d', is then assigned under each column as shown in table 9.

Experiment Runs	Si	Cu	Mg	Zn	Mn	Sn	Cr	Ni
1	0	0	0	0	0	0	2	2
2	1	0	0	3	2	3	0	2
3	2	0	3	2	3	1	3	0
4	3	3	2	0	0	1	1	0
5	0	3	2	0	3	3	2	3
6	1	3	1	3	0	3	3	1
7	2	3	1	1	3	1	0	2
8	3	2	2	3	1	0	2	3
9	0	2	3	2	1	0	0	2
10	1	2	1	3	3	0	1	0
11	2	0	3	0	2	3	1	1
12	3	1	2	2	1	2	2	3
13	0	1	3	2	0	2	1	1
14	1	2	0	1	2	2	3	0
15	2	1	1	1	2	0	3	3
16	3	1	0	1	1	2	0	1

Table 9: Shows all the factor level assigned to the columns.

All factor level is given an influence value based on the percentage limit of the allowable alloying percentage of each alloying element on aluminium illustrated by table 10. Total influence of each entry is multiplied with the factor levels to obtain the total weight of each alloying elements, 'n' which totals to 160 entries.

s

	Factor levels				
Elements	**0**	**1**	**2**	**3**	
Si	0	2	4	6	wt%
Cu	0	2	4	6	wt%
Mg	0	2	4	6	wt%
Zn	0	2	4	6	wt%
Mn	0	0.2	0.4	0.6	wt%
Sn	0	1	2	3	wt%
Cr	0	0.2	0.4	0.6	wt%
Ni	0	1	2	3	wt%

Table 10: Shows the influence value of each factor level of a variable.

Table 11 shows the completed orthogonal array done for experimental run 7, 8 and 9 after combining table 10 and table 9, full table with all 16 experimental runs is available in the appendix. Titanium is added as a grain refiner and kept constant at 1% for all experiment runs.

Experiment Runs	**Al-50Si(g)**	**Pure Cu (g)**	**Pure Mg (g)**	**Pure Zn (g)**	**Al-60Mn (g)**	**PureSn (g)**	**Al-10Cr (g)**	**Al-19Ni (g)**	**Al-3Ti-B (g)**	**Remaining Aluminium (g)**
7	80	60	20	20	10	10	0	105.26	1	693.736842
8	120	40	40	60	3.33	0	40	157.89	1	527.78
9	0	40	60	40	3.33	0	0	105.26	1	750.406842

Table 11: Taguchi's Orthogonal Array developed for experimental run 7,8 and 9.

4.3 Casting Process

Preparation was done before casting where the metals were cut to its respective weight. Before beginning a check of the sectioned metal are done to ensure there are no missing metals and all metals are of correct weight. Next, casting equipment was painted. Ensuring molten metal won't stick to the surface of equipment, the tongs, moulds, crucible and stirrer, using boron nitride

coating. The size of crucible and moulds selected must be the same for consistency and be able to handle 1 kg of molten metal. A 10% disparity between the stated percentage weightage of orthogonal array and actual alloying elements percentage added during casting is allowed for the inaccurate sawing machine.

First the metals, crucible and mould are preheated in the preheating furnace at 200 °C for an hour or two to remove all moisture. Moisture must be removed during casting to prevent the moulds and crucibles from cracking. After, the aluminium in clay crucible is transferred to magnesium furnace which has been left to heat up to a temperature of 780 °C, selected based on the melting point of aluminium which is 665 °C. [34] When the aluminium melt, the alloying elements in preheating furnace is can then be taken out to be added into molten aluminium. Alloying elements is slowly added at intervals of 10 to 30 minutes to allow the alloying element to properly dissolve into aluminium. Another 30 minutes of waiting time is required after additions of all alloying elements to allow for all alloying elements to dissolve. Addition of alloying elements must also only be done at temperature above 710 °C, to allow for the metal to partly melt upon addition into molten metal. Lastly the molten alloy is will be poured into coated moulds which has been preheating furnace. Mould cavity should be overfilled to compensate for aluminium alloy which has high degree of shrinkages during cooling and solidification. Excess liquid metal is then poured into a separate mould of similar size which is preheated as well. Pouring temperature should always be at 760 °C. Size of crucible and moulds and pouring temperature all kept constant to reduce the variables in experiment. The molten aluminium alloy was then left to solidify in mould cavity by air cooling at room temperature.

Magnesium is always added last due to its reactivity, a special stirrer with umbrella-shaped head is required to push the magnesium to the bottom of crucible to ensure the magnesium is not floating on the molten metal which can cause reaction to oxides in ambient air. Tin should not be preheated with other alloying elements as it has low melting point and will melt during preheating.

4.4 Methodology and Justification of Heat Treatment Process

Inferring from figure 8 of literature review, finding the optimum solution heat treatment temperature is a balancing act of maximising the heat treatment temperature with longest holding time possible. The best solution treatment temperature and holding time was determined by trial and error approach. Educated guess stemming from analysis of previous results will substantiate

following trials until the optimum temperature and holding time capable of dissolving the most intermetallic phase without partial melting is ascertained.

Each individual aluminium alloy has a different optimum solution treatment temperature because of different alloying constitutions. Temperatures for each run were selected based on the melting point of pure aluminium at 665 °C and assumed eutectic point of the aluminium, lowest liquidus temperature of each alloy. [34] Usually, aluminium alloy in references to journals have eutectic point between 500 °C to 600 °C. The result yielded from initial solution treatment will be corroborated using microstructure analysis to optimise subsequent trial runs.

Justification for trial and error approach is the amount of alloying element renders the use of conventional binary phase diagram not practical to determine the eutectic temperature of the alloy. An alloy with two or more alloy is considered eutectic alloy with eutectic phase diagram. But to properly represent all the alloying constitutions added, phase diagram with intermetallic compound and intermediate phase must be constructed. Not all intermetallic phases are expected to be dissolved because most alloys do not have complete solid solubility as stated in literature review. Effort will not be made to dissolve the higher temperature intermetallic particles as it will result in distortion and partial melting of other intermetallic particles of the alloy that homogenise before.

Based on aforementioned literature review, temperature of aging is usually done around 115 °C to 200 °C with up to 3 days of holding time. This theoretical knowledge is supplemented by scientific article by Aytekin Polat et al, which perform aging at temperature of 160 °C, 180 °C and 200 °C couple with aging time up to 80 hours or just over 3 days. Consideration is given to aging process of this particular journal as the alloy tested in the journal consisting of Mg, Si, Cu, Mn, Fe, Cr and Zn is similar in composition albeit in lower percentage to the alloys casted for this thesis. Deriving from these knowledge, aging temperature of 160 °C, 180 °C, 200 °C and 230 °C and aging time of 6 days is selected for this thesis. Additional aging temperature 230 °C and aging time of up to 6 days is implemented to ensure proper coverage for all possible cases of aging curve.

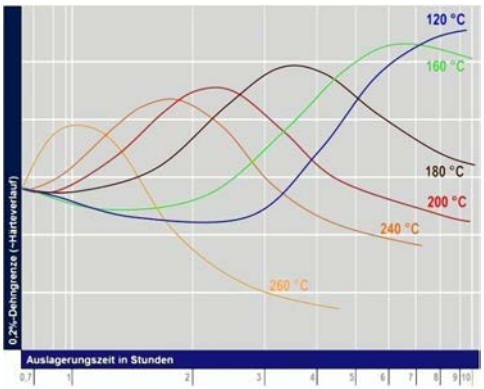


Figure 16: Possible shape of aging curve. [57]

From figure 16, there are possibility of peak hardness obtained after a longer aging time such as the blue aging curve. Aging up to 6 days instead of the conventional 3 days will ensure that any alloy which exhibits such aging trends to be accounted for. Aging at higher temperature beyond optimum will not yield any beneficial results. Temperature of 230 ° is added to validate that optimum aging of all alloy sample is indeed below 200 °C.

4.4.1 Solution Heat Treatment Process

The casted aluminium alloy is divided into smaller size in the shape of pie. As the casted alloy is in the shape of cylindrical blocks, pie shape is selected to ensure the properties of aluminium alloy casted can be thoroughly tested as there are reasonable doubts that the properties of the aluminium alloy core and outside surface will have similar properties due to different rate of cooling.

With the pie shaped samples, solution treatment can then begin. There are 4 initial solution heat treatment temperature selected for solution treatment at 520 °C, 530 °C, 550 °C and 570 °C with 2 hours holding time. To solution treat each sample, a steel container is partially filled to half with fine sand and a steel netting with aluminium alloy sample is strategically situated on the sand to prevent contact with other alloy pieces and steel netting. The remaining space in the steel container is filled with fine sand and topped out with carbon pieces in a 90% sand and 10% carbon ratio. Sample is surrounded by sand during solution treatment to prevent surface of sample to come in contact with air and also allow for better distribution of heat to ensure an even heating is applied onto the samples. Additional steps to prevent oxidation was done by placing carbon pieces which is more reactive to oxygen on top of the container to oxidise in place of alloy samples. The container is then closed with a lid and placed into the middle of furnace with thermocouple stuck into the container through a hole on the cover as close to sample as possible, allowing for temperature of furnace to be adjusted accordingly to compensate for the discrepancies of furnace temperature and sample temperature.

Best solution treatment temperature is determined by performing microstructure analysis on the solution treated sample. Following the identification of the best solution treatment temperature, 20

additional samples for each experimental run is solution heat treated as well at the respective optimum temperature and holding time. All failed iteration leading to the final optimum solution treatment is chronicle in appendix.

4.4.2 Age Hardening Process

Heat treatment is only complete if solution treatment is followed by age hardening causing the nucleation of fine precipitate from alloying elements which promotes the hardness of steel. Again, there are 4 aging temperature of 160 °C, 180 °C, 200 °C and 230 °C. The aging time varies from 15 mins up to 6 days over an interval of:

Aging Times									
0	15 mins	30 mins	1 hour	1 hour 30 mins	2 hours	3 hours	5 hours	10 hours	12 hours
15 hours	18 hours	24 hours	36 hours	48 hours	60 hours	72 hours	96 hours	120 hours	144 hours

Table 12: Aging times selected for solution treated aluminium alloys.

Alloys heat treated is placed in furnaces set to desired temperature. They are held at these specific temperatures for a period of time as specified in table 11 before removal for air cooling. To ensure sample is not compromised by further natural aging or external unaccounted for factors, hardness test is done on alloys within a day of removal from furnace or kept at low temperature in fridge to prevent further microstructural changes. All hardness of alloys is compiled to plot aging curve for each alloy to identify underaging, peak aging and overaging.

4.5 Vickers's Hardness Test Methodology

All as-cast, solution treated, and age hardened sample hardness was tested using Vickers's hardness test. The load for each test is kept consistent at 2 kgs with 10 s dwell time. A load of 2 kg was chosen because hardness of aluminium and aluminium alloy is relatively lower in value and sample is not very thick with about 0.3 cm to 0.5 cm thickness on average. It is assumed that 2 kg load is as large as possible, preventing any errors due to variation between surface layer. Aged alloy with higher hardness may require a higher load of 3 kg with similar dwell time to get sufficiently large indents for easier analysis. Measurements are taken from the front, centre and along the edge, to ensure there are no discrepancies in properties across surface of, samples arising from an error in heat treatment and casting. At least 8 readings are taken and averaged out for each sample to obtain

more consistent and accurate result. Highest and lowest readings taken is ignored and was not averaged to prevent the final hardness value to be skewed or biased.

Vickers's hardness test requires the sample to be indented for testing and directly effects the results as measurement of both edges has to be taken for a hardness value to be obtained. All pie shaped samples top and bottom surface is grinded on the grinding machine with 120-grade roughness grinding paper to roughly get a flat and more even surface. Grinding with 120-grade roughness grinding paper can be obsolete if sample is mounted, where the surface will be flat and will only require grinding with 600-grade roughness grinding paper. Any uneven surface will distort the shape of the indentation which may enlarge or make the indentation smaller. Grinding and polishing for some cases also removes contaminants from surface of the sample which can cause inaccuracies in reading as the diamond indenter is a very sensitive instrument. Indentations for reading are not to be done on or near porosity or previous indentation as a bigger and deeper indentation than normal conditions will happen.

4.6 Microstructure Analysis Methodology and Reasoning

To correlate the result of hardness test and observe the effect of heat treatment on the sample, microstructure analysis is done. Surface of sample after casting and heat treatment is not clean and has porosity in some cases, preventing microstructure of the sample from showing. For the microstructure of the sample to show, all samples undergo grinding, polishing and etching in some cases. Grinding is done at 320, 600, 1200 and 4000 grade of roughness grinding paper to remove a small layer of surface of the sample. The grinded surface is then polished to clean the surface of the sample using mol, nap- b and chem cloth. After each grinding and polishing process, the sample was thoroughly cleaned with distilled water and ethanol. Any defects or scar on the surface of sample after grinding was grinded again with a lower grade roughness paper to remove the defects. A few drops of chemical were dropped onto the surface of the sample to etch for a while. All samples undergoing microstructure analysis was mounted to allow for automatic grinding on machine and easier handling when etching. Sample which exhibits high porosity is ultrasonic cleaned to remove impurities residing in the pores and crevices.

Etching of samples is done with 2%Nital solution, which is commonly used for aluminium alloy. While not the most potent etchant as etching can be done with up to 10%Nital solution or even more potent chemicals such as hydrofluoric acid. It is not needed however, as polishing done on the surface of sample is throughout enough. In most cases polishing up to chem grade will reveal

the microstructure of the sample just fine. Some solutions poses safety risk such as above 5%Nital is actually highly flammable liquid and should be avoided if possible. [35]

Etching time is dependent on the sample itself and differ from sample to sample due to the different constituents of each alloy. For sample 7 and 9, the etching time was about 90 seconds while sample 8 has a much shorter etching time of 30 seconds. All etching time for the samples was determined by closely monitoring any dulling in the surface of the polished sample. This method is fairly inaccurate as usually bubbles will form on the surface of the sample once indicating that proper etching has been done on the surface but due to the low potency of 2%NiTal solution, trial and error approach has to be taken. Any over etching will require the sample to be grinded and polished again.

Microstructure images of as- cast, solution treated, and age hardened aluminium is viewed and taken to be inferred. Analysis of solution treated alloy is especially important for the identification of optimum solution treatment temperature and time by comparing the amount of phases homogenised compared to the as- cast microstructure.

4.7 Analysis by Orthogonal Array

Once peak hardness is identified for each sample after age hardening, orthogonal array analysis can begin. We are only interested in the peak hardness, all other aging temperature possessing lowers peak hardness will not be taken into account during analysis. Referring to the aim of the experiment, effect of variables to be studied by this thesis is the alloying element effect on the aging hardness obtained, not the effect of aging temperature on the hardness obtained.

The peak hardness obtained after heat treatment is compiled in tabular form alike table 10 with an additional row for peak hardness is added. Hardness of each experimental run can be compared and effect of dominant alloying element in alloy can be studied. For example, if sample 4 is determined to have very high hardness after age hardening, we can then refer to find the dominant alloying element for sample 4.

Analysis is further detailed by the respective alloying elements and respective percentage to the hardness obtained to corroborate the findings. Hardness of alloy with same alloying element and its percentage is average and plotted by the percentage. Say for 0 factor level of silicon is added to alloy 3, 4, 8 and 9. The hardness of these alloys will be averaged and so forth for 1, 2 and 3 factor

level. A plot to study the hardness obtained with their respective alloying percentage can be studied individually.

5. RESULTS

5.1 Sample 7: Al-4Si-6Cu-2Mg-2Zn-0.6Mn-1Sn-2Ni-1Ti

5.1.1 Sample 7: Casted Alloy and Microstructure

Experimental Run 7	
	

Table 13: Images of resulting casting for Al- 4Si- 6Cu- 2Mg- 2Zn- 0.6Mn- 1Sn- 2Ni- 1Ti.

Metal with deviation from orthogonal array weight.		
Metal	Calculated weight	Actual weight
Aluminium	693.7	695
Zn	20	22
Ni	105.26	106.5

Table 14: Deviation of weight for experimental run 7.

The total weight of the casted alloy for experimental run 7 is 967 g. Casted alloy experience small amount of metal burnt off and oxidisation during casting. The weightage of alloy added is not exact as well, difference from calculated and actual weight added for casting is noted on table 14. Shrinking experience by the ingots are negligible.

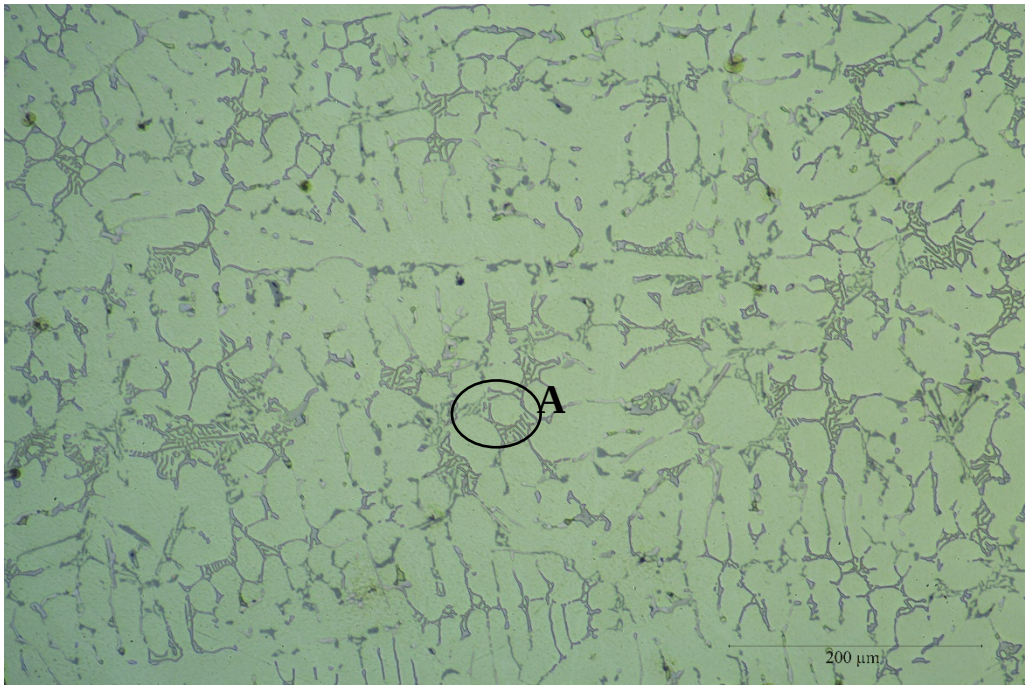


Figure 17: Microstructure of as- cast for Al- 4Si- 6Cu- 2Mg- 2Zn- 0.6Mn- 1Sn- 2Ni- 1Ti

Microstructure of as-cast sample 7 shows typical eutectic microstructure along high concentration of alpha phase of aluminium, α-Al grains, typical of conventional casting. The α-Al grains are highly homogenised and large with features akin to enlarged dendrites. Correlation of observation is found to journals by Helmut Kauffman et al. and Shujian Cheng et al. casting at above aluminium liquidus temperature of 625 °C will result in highly rosette and dendritic α-Al grains as in this case where casting is done at temperature of 730 °C. [50] [51] Casting at lower temperature nearer to liquidus temperature results in spheroidise and smaller α-Al grains. [50] The dominant secondary phase is eutectic phase. Eutectic phase of sample 7 seems to have a varied shape between conventional eutectic phase or chinese script.

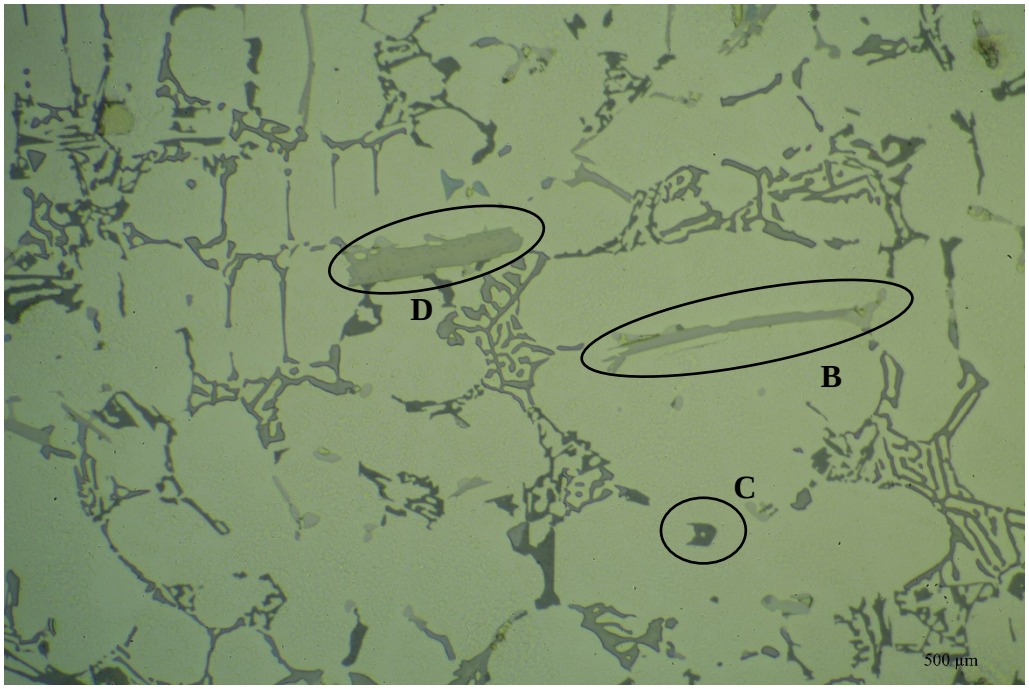


Figure 18: Sample 7 as- cast microstructure 500x magnification.

In figure 16(A), shows the rounding and coalescence of constituents, most likely silicon, between the interdendritic phase. [72] Another feature is two separate needles intermetallic phase of white colour marked 'B' in figure 17. Patches of undissolved intermetallic precipitate in platelets shape, 'D', and random shaped intermetallic marked as 'C' in shown respectively figure 17, distributed in small number throughout the microstructure. Random combination of alloying elements and base metal, exemplified by Cu and Mg phases, requires solution treatment to dissolve.

Sample 7: As- Cast Vickers Hardness (HV)				
80.5	86.3	83.7	86.2	82.4
Average Hardness: 83.8				

Table 15: Sample 7 average as- cast hardness.

The average as-cast hardness of alloy 7 shows intermediate hardness in comparison to common commercialised aluminium alloy of 1xxx to 7xxx series with hardness in the range of 30 to 160 HV. [62]

5.1.2 Sample 7: Solution Treatment Results

Optimum solution treatment for sample 7 is at 540 °C coupled with a holding time of 3 hours.

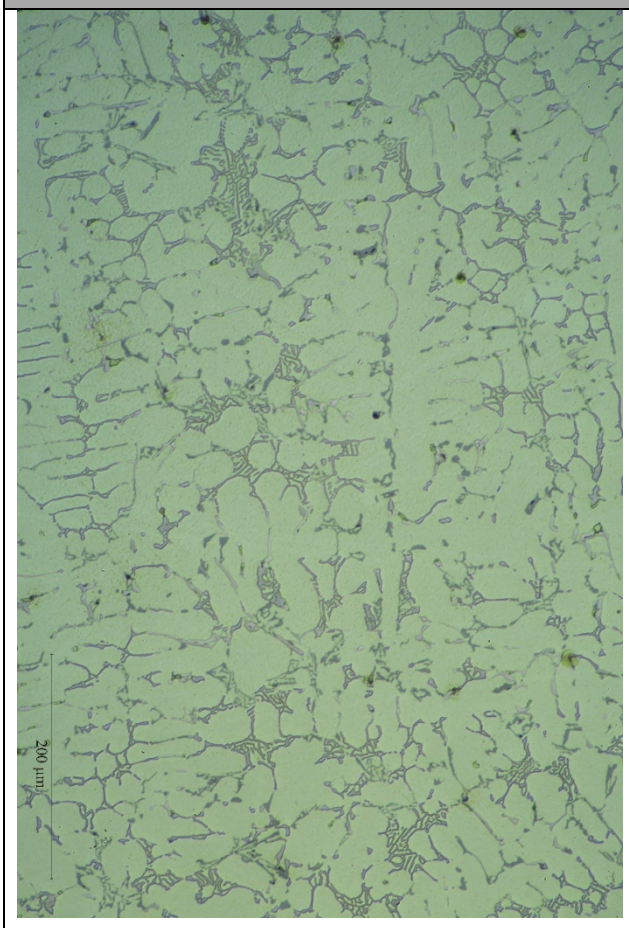
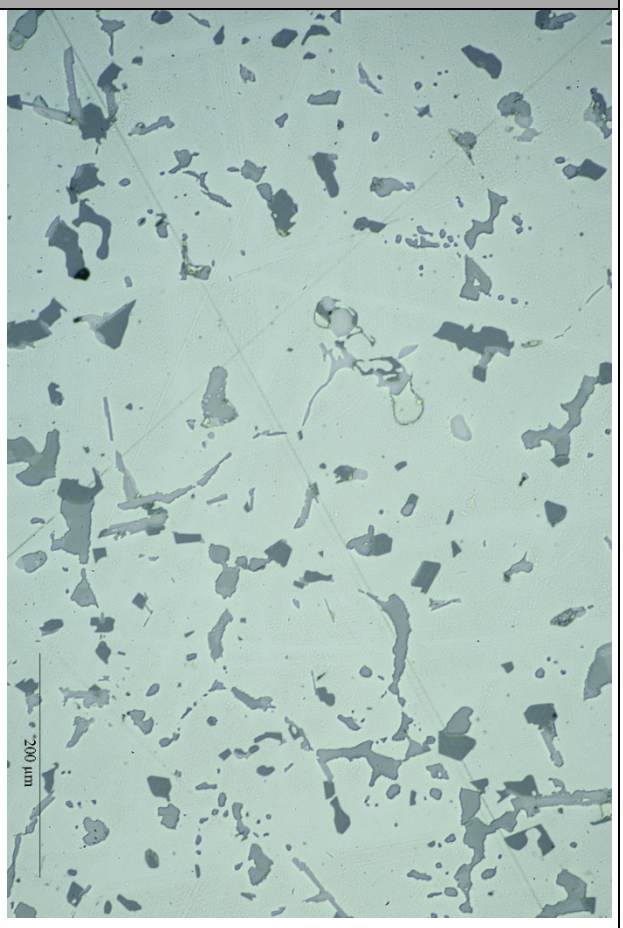
As- Cast Sample 7	Solution Treated Sample 7
	

Table 16: Compares sample 7's as- cast and solution treated microstructure.

From table 16, after solution heat treatment, alloy 7 phases and intermetallic compound has mostly dissolved. One changes at a glance are the eutectic phase which has dissolve completely into the alpha aluminium phase. The platelets shaped and needle-shaped intermetallic compound in figure 18 has are also dissolved as well. In place are coarsened intermetallic compounds amongst the alpha aluminium phase. Bigger intermetallic particles observed are from movement of atoms of excess undissolved intermetallic particle from as-cast condition that begin to cluster and coarsen. Referring to the orthogonal table in section 4.2, sample 7 exhibits high percentage of copper, zinc and silicon in comparison with other alloying elements. A journal by E. TILLOVÁ and M. PANUKOVÁ, highlights similar coarsening effect seen when there is high percentage of silicon, zinc and copper in alloy matrix. [63] The composition in the journal is similar to the alloys of this thesis as well with the main difference being the alloying percentage. Another similarity of the

journal alloying composition has higher silicon and copper content in relation to other alloying elements similar to that of alloy 7. As such it can be theorised that coarsening effect seen is related to presence of silicon and copper in the alloying constituents, although Scanning Electron Microscope or other advance form of microanalysis technique should be performed to be sure.

5.1.3 Sample 7 Aging Curve and Aged Hardness Results

Previously mentioned in section 4.7, orthogonal array analysis requires only the sample which produce the highest hardness after age hardening. Hence only the peak hardness aging curve and its respective microstructure will be discussed for sample 7, 8 and 9. Other aging curve and full data logs are available in the appendix section along with corresponding standard deviation for each hardness results. Standard deviation is added to the aging curve to ensure the integrity of result obtained.

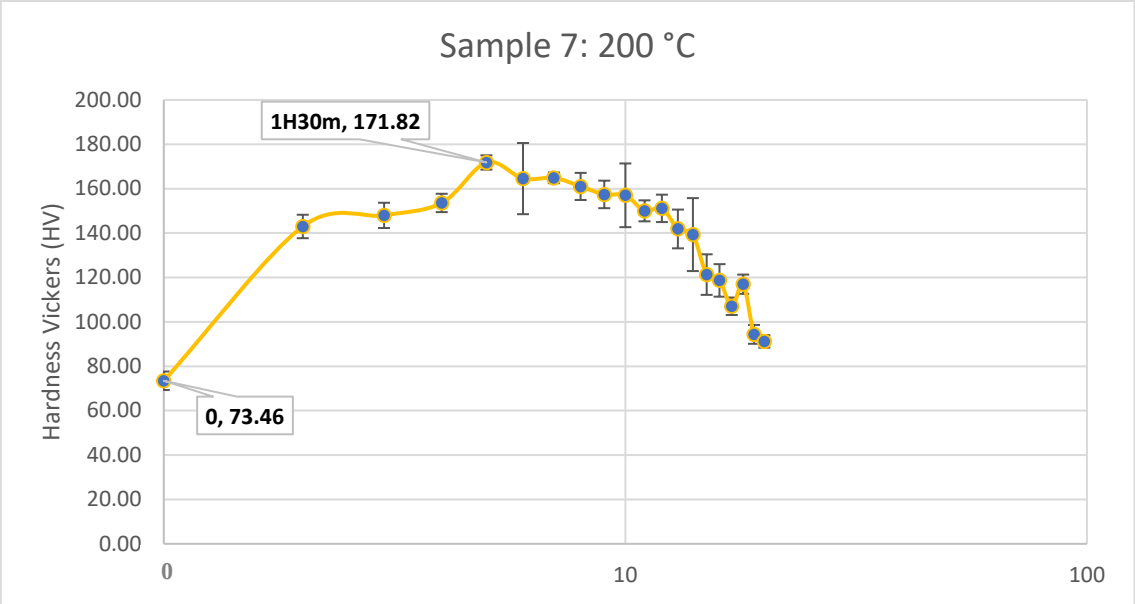


Figure 19: The plotted aging curve which yields peak hardness for alloy 7 after aging at 200 °C.

The highest hardness logged for sample 7 was obtained after aging at 200 °C with 1 hour 30 minutes aging time. Peak hardness obtained is 171.82. an increment of about 90 HV from original as-cast hardness of 83.8. This indicates that heat treatment was carried out successfully and sample 7 respond well to heat treatment. Hardness at 0 in figure 19 is hardness obtained after solution treatment on alloy 7. Hardness reading at 0 in figure 19 is solution treated hardness. Error bars on the aging curve signify the standard deviation of hardness readings. Hardness value obtained can be assumed to be quite consistent with almost no large deviation in error bar signifying no large outliers in reading.

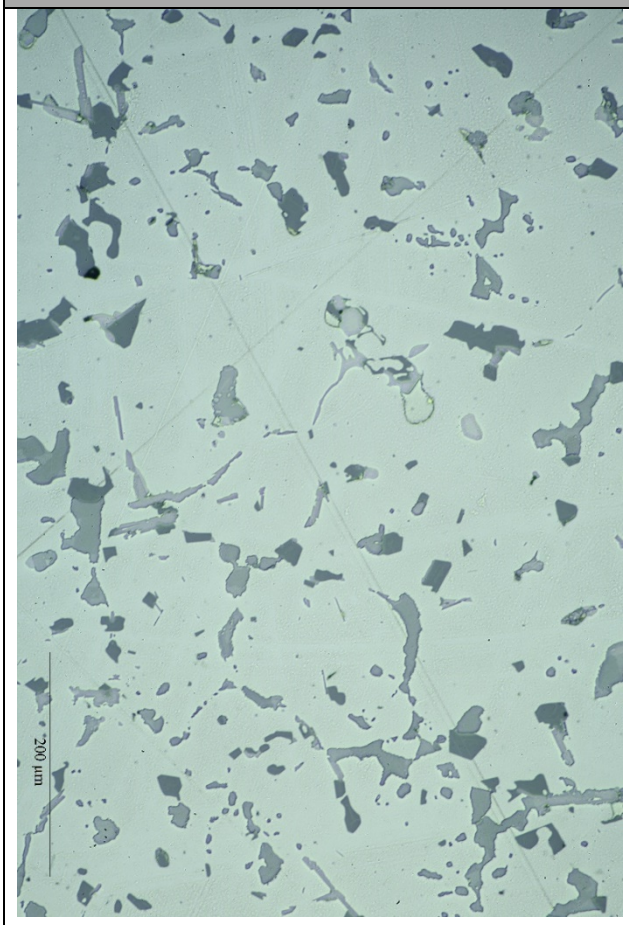
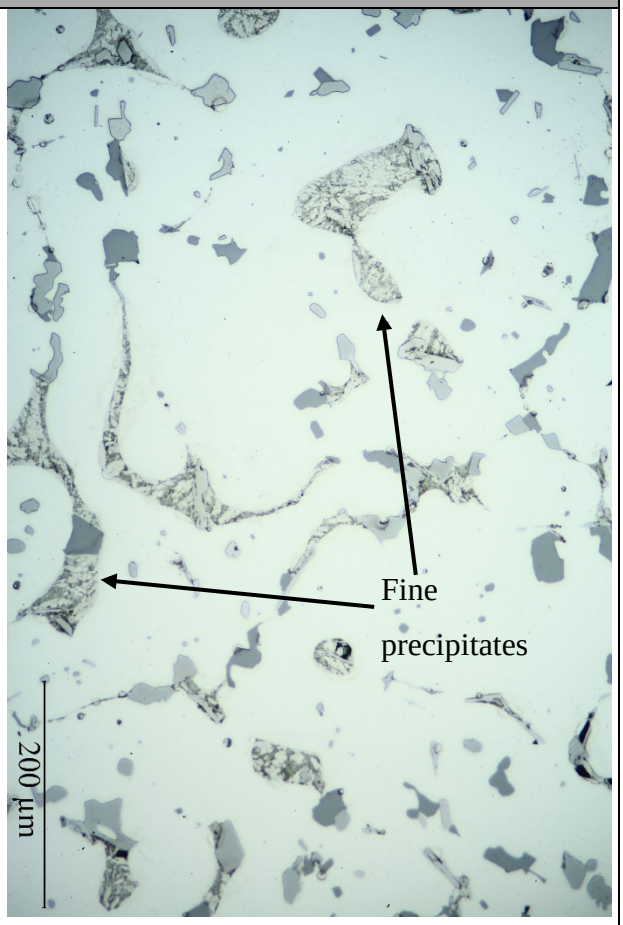
Solution Treated Sample 7	Peak Aging Microstructure Sample 7
	

Table 17: Compares sample 7 solution treated and after aging microstructure.

Identifiable difference between the solution treated microstructure and after aging microstructure is the nucleation of fine precipitates on the α-phase aluminium and around the previously haven't dissolved intermetallic compounds. Number of intermetallic compounds observed is noticeably less and is assumed to have nucleated as fine particles after aging. Formations of these fine intermetallic particles corroborate to the increase in hardness obtained after aging as these fine precipitate will impede dislocation movement and thus the hardness of alloy according to literature review.

5.2 Sample 8: Al-6Si-4Cu-4Mg-6Zn-0.2Mn-4Cr-6Ni-1Ti

5.2.1 Sample 8: Casted Alloy and Microstructure

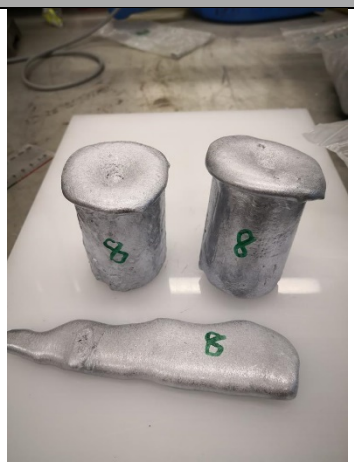
Experimental Run 8	
	

Table 18: Images of resulting casting for Al- 6Si- 4Cu- 4Mg- 6Zn- 0.2Mn-.4Cr- 6Ni- 1Ti.

Metal with deviation from orthogonal array weight.		
Metal	Stated weight	Actual weight
Ni	157.9	155.0
Zn	60	58
Al-50Si	120	119

Table 19: Deviation of weight for Al- 6Si- 4Cu- 4Mg- 6Zn- 0.2Mn-.4Cr- 6Ni- 1Ti.

The difference in weight calculated and added for casting is tabulated on table 18. Sample 8 experience slight burnt off and oxidising as well. On closer inspection upon sectioning sample 8 into smaller pieces, porosity is prominent along the core of sample 8. Porosity on alloy 8 can be attributed to the alloying constituents of sample 8, a more controlled casting process is required if better casting is desired instead of conventional casting with turbulent pouring of molten alloy into mould.

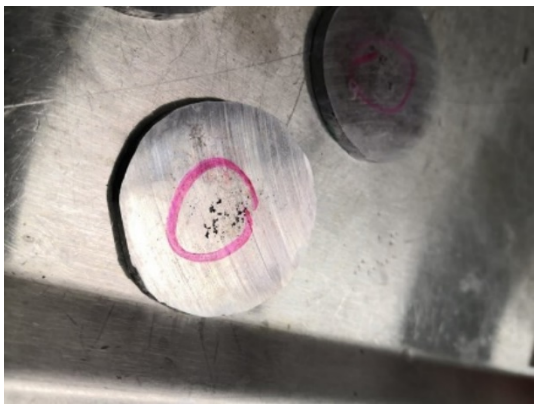


Figure 20: Porosity at core of sample 8.

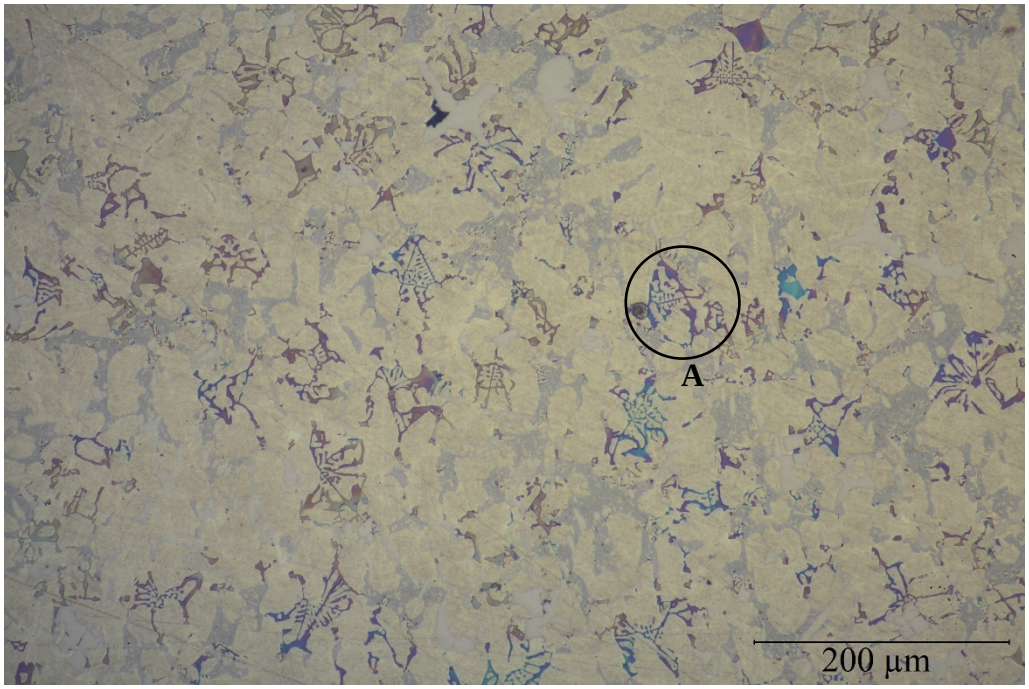


Figure 21: Microstructure of as- cast for Al- 6Si- 4Cu- 4Mg- 6Zn- 0.2Mn-.4Cr- 6Ni- 1Ti.

Similar to sample 7, microstructure of as- cast for ally 8 shown in figure 20 is dominated by alpha phase alloy of aluminium, α-Al grains. There are presence of coarse eutectic phases as well, but eutectic phase is predominantly in the shape of chinese script, figure 20(A).

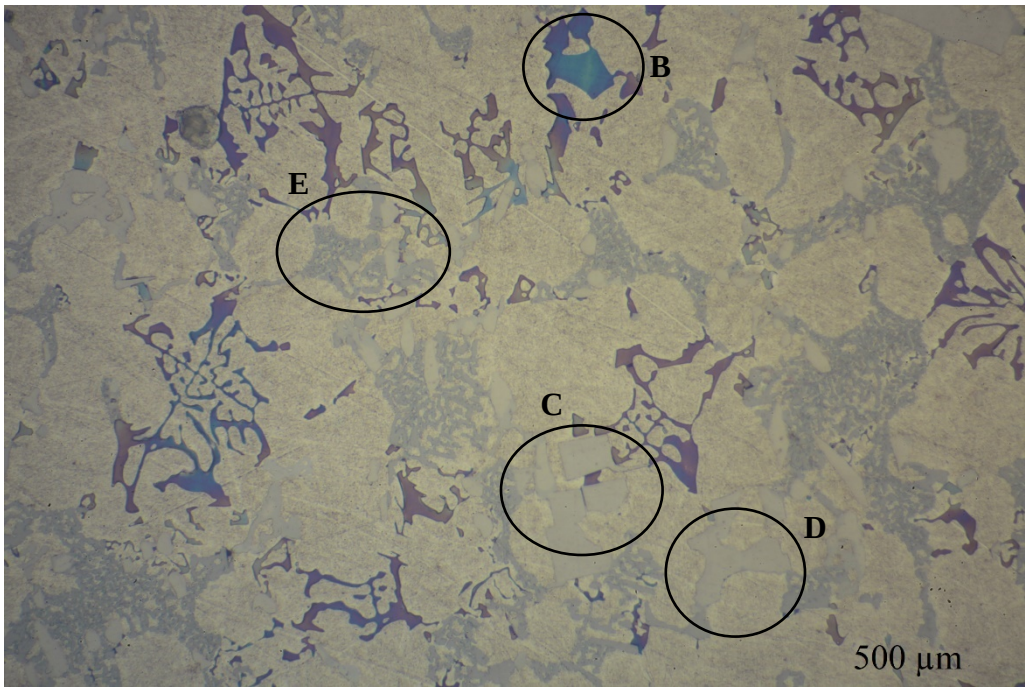


Figure 22: Shows sample 8 as-cast microstructure at 500x magnification.

In place instead are more intermetallic phases represented by colourful intermetallic compound, figure 21(B), platelet-shaped white intermetallic phase, figure 17(C) and dendrite shaped white intermetallic phase, figure 17(D). Increase in type of undissolved intermetallic phase can be related to the higher percentage of alloying element with tendency to combine and form non-dissolvable intermetallic compounds such as zinc, copper, magnesium, silicon and chromium. The intermetallic compound 'B', 'C' and 'D' will ideally dissolve and form fine precipitate after heat treatment. On higher magnification, alloy 8 exhibits another eutectic phase which is very fine marked in figure 21(E).

Sample 8: As- Cast Vickers Hardness (HV)				
154.5	165.1	149.9	158.8	156.6
Average Hardness: 157				

Table 20: Sample 8 average as- cast hardness.

The average as-cast hardness of alloy 8 shows very high hardness at 157 HV in comparison to common commercialised aluminium alloy of 1xxx to 7xxx series with hardness in the range of 30 to 160 HV. [62]

5.2.2 Sample 8: Solution Treatment Results

The optimum solution treatment temperature identified for sample 8 is 540 °C with 3 hour holding time as well.

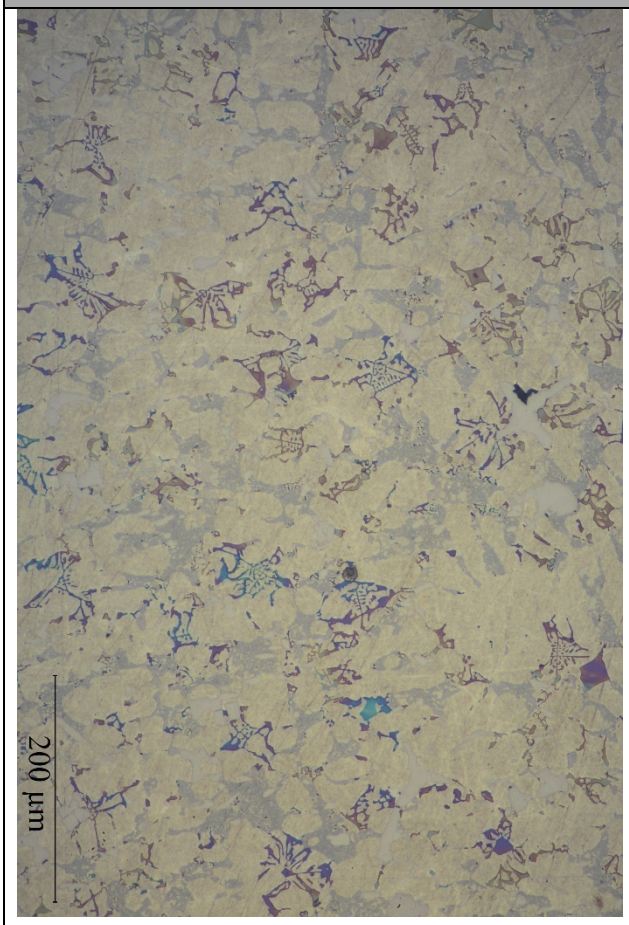
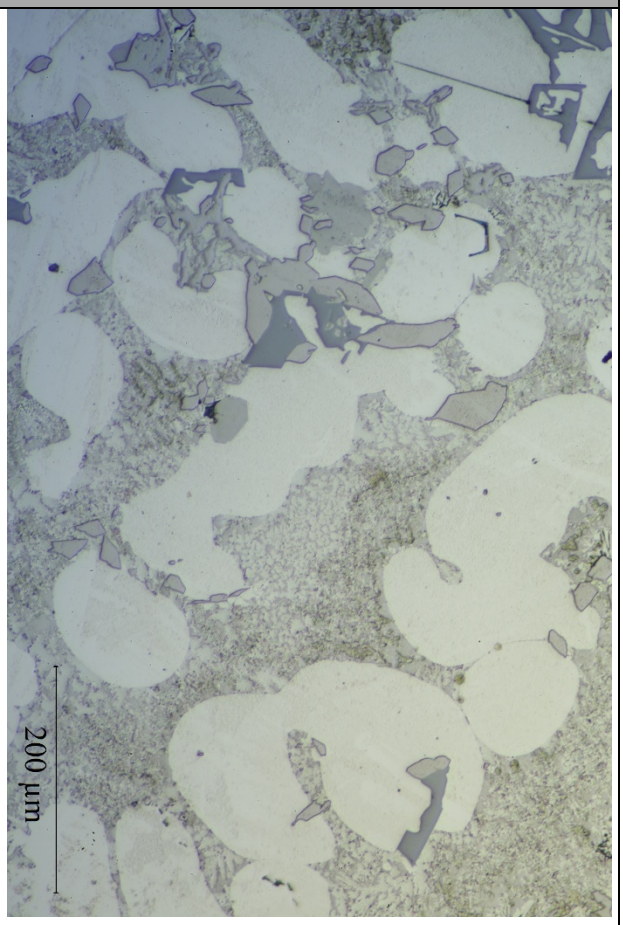
As- Cast Sample 8	Solution Treated Sample 8
	

Table 21: Compares sample 8's as- cast and solution treated microstructure.

Comparing the as-cast and solution treated microstructure of alloy 8, the intermetallic phase seen before has spherodised and clustered into fine intermetallic compound. There are still a little leftover undissolved intermetallic compound that exhibits similar coarsening observed in alloy 7 as well. A check on the orthogonal array in section 4.2 shows alloy 8 has high amount of silicon, zinc and copper percentage such as alloy 7. Again, this shows that coarsening effect of intermetallic compound of alloy can be theoretically related to high percentage of silicon, zinc and copper added as proven by the journal by E. TILLOVÁ and M. PANUKOVÁ. [63] More advanced microscopy analysis is to be done to prove these assertions but it is expected that intermetallic compound of journal and alloy sample 8 while showing coarsening trend will have slight difference in minor makeup of the intermetallic compound from other alloying elements.

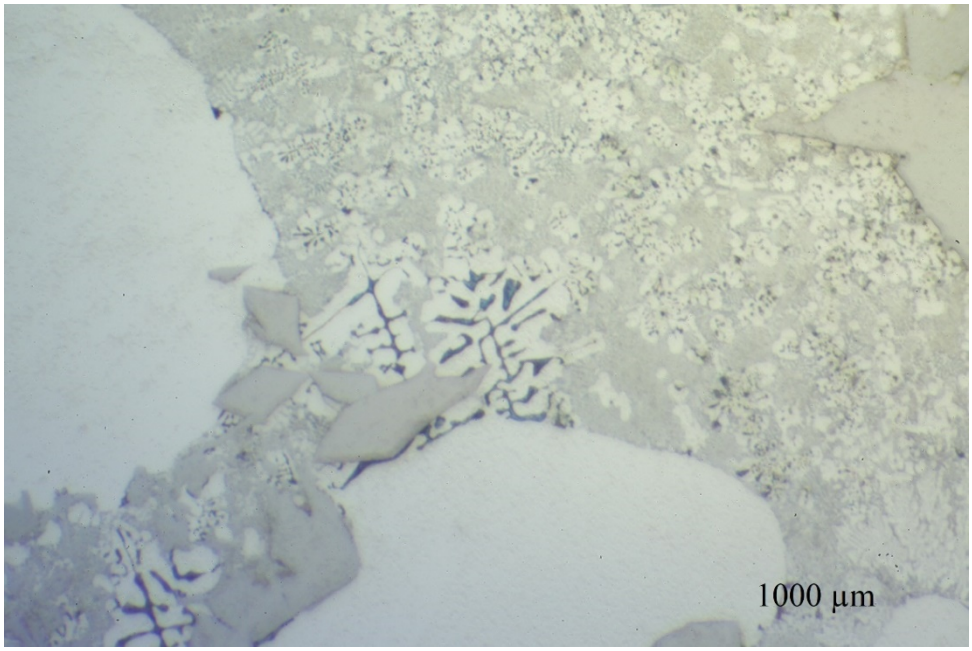


Figure 23: Solution treated alloy 8 showing fine eutectic phase.

Microstructure analysis with higher magnification at 100x shows that in between spherodise intermetallic compounds are partially dissolved and clustered eutectic phase. These fine eutectic phases retain the chinese script shape. It can be inferred that if solution treated for a longer time, the eutectic phase will fully spherodise and dissolve. Attempts to do so, however, result in partial melting and distortion in alloy 8 as other phases of alloy 8 perhaps have lower liquidus temperature compared to the eutectic phase of alloy 8.

5.2.3 Sample 8: Aging Curve and Aged Hardness Results

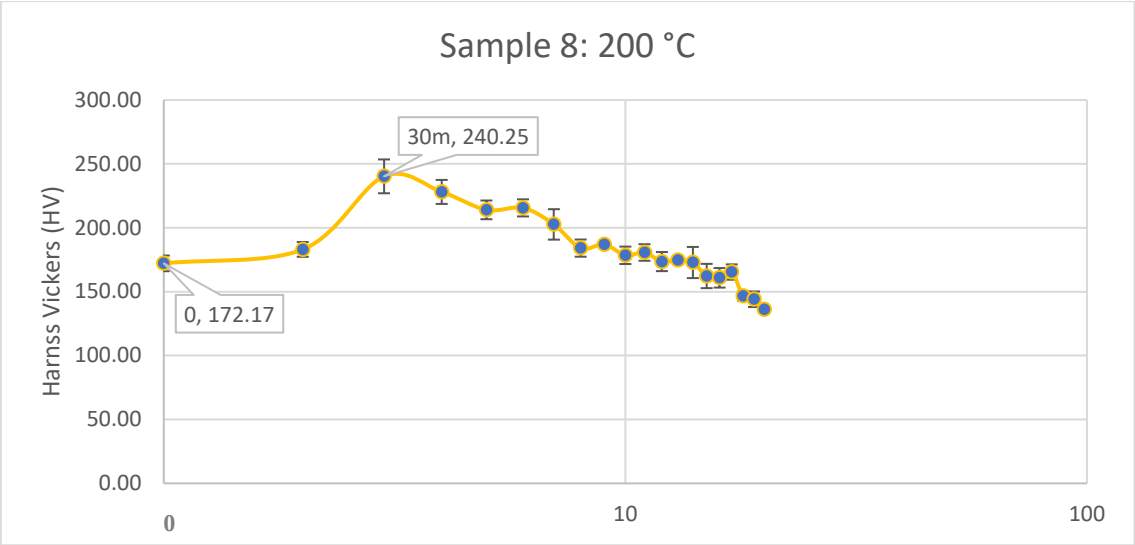


Figure 24: The plotted aging curve which yields peak hardness for alloy 8 after aging at 200 °C.

Sample 8 produces the alloy with highest peak hardness among sample 7, 8 and 9. Peak hardness of alloy 8 is obtained after aging for 30 minutes at temperature of 200 °C. Hardness increased from the original as-cast condition of 157 to 240, an over 80 HV increment in hardness indicating proper heat treatment has been carried out and sample 8 respond well to heat treatment as well. Hardness at 0 in figure 24 is hardness obtained after solution treatment on alloy 7. Hardness at 0 in figure 24 is hardness obtained after solution treatment on alloy 8. Error bars on the aging curve signify the standard deviation of hardness readings. Hardness value obtained can be assumed to be quite consistent with no large deviation in error bar signifying no large outliers in reading.

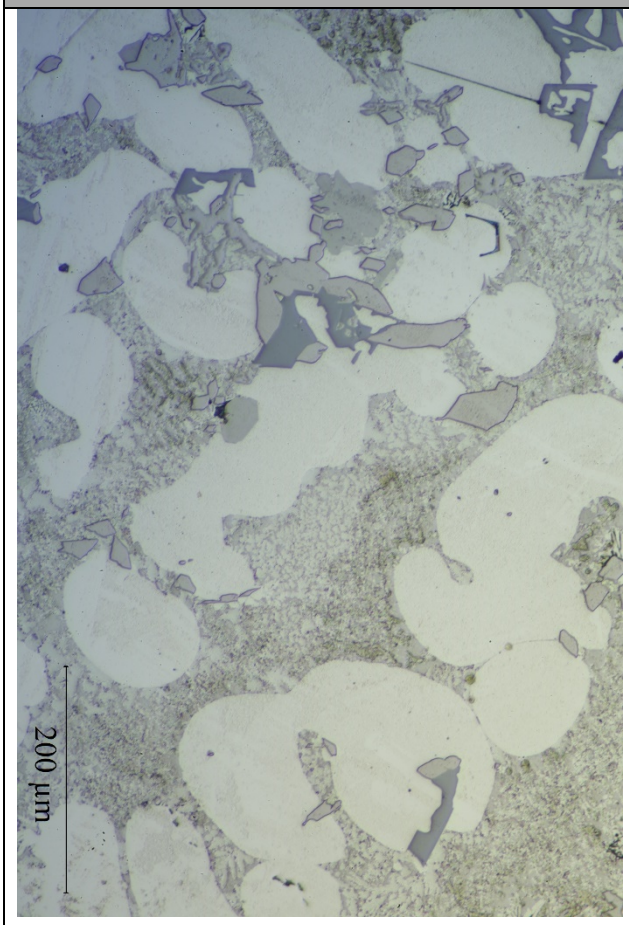
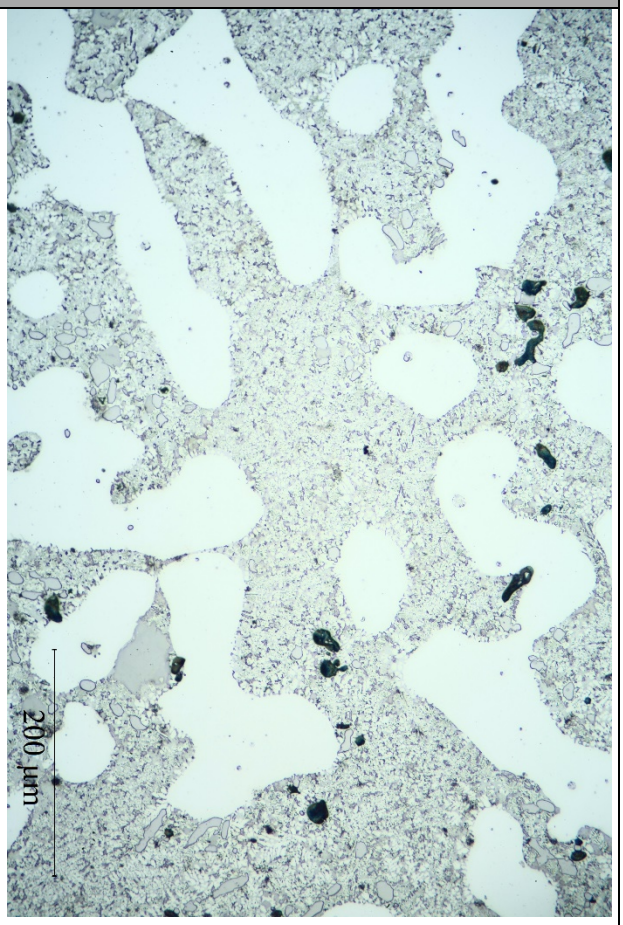
Solution Treated Sample 8	Peak Aging Microstructure Sample 8
	

Table 22: Compares sample 8 solution treated and after aging microstructure.

The microstructure of alloy 8 after aging shows that all the previously undissolved intermetallic compounds has dissolved into the alpha phase and nucleated into fine precipitates. The concentration of cluster of fine intermetallic compound is also higher than previously seen for solution treated samples. Grains of α-phase aluminium is now more clearly defined, entrapped by the fine intermetallic precipitates.

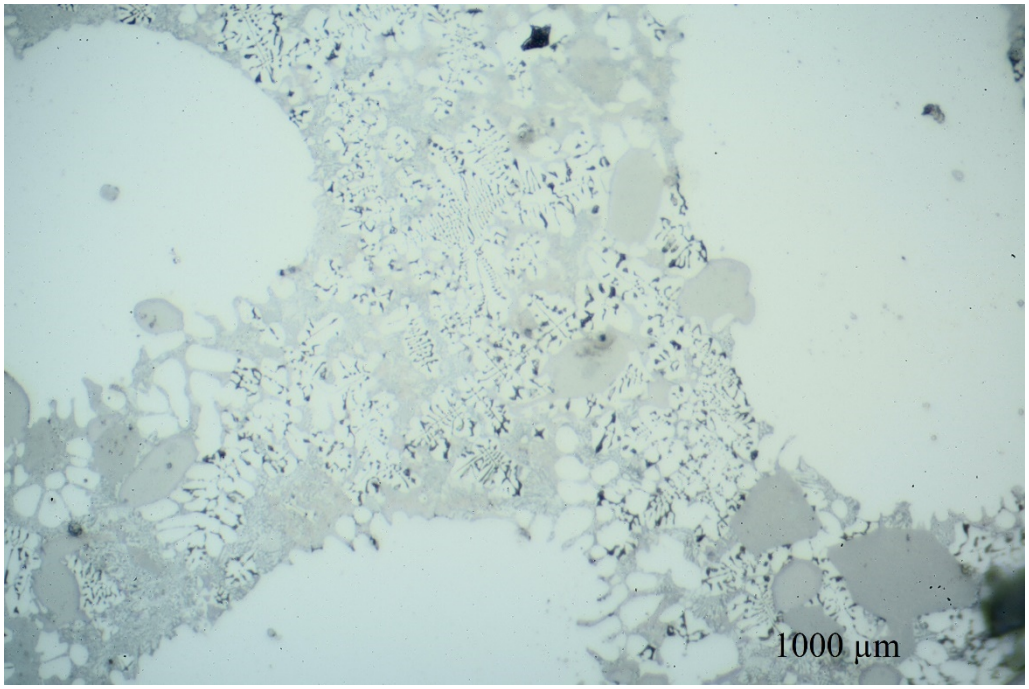


Figure 25: Peak aging microstructure of sample 8 at 100x magnification.

Upon closer inspection, the fine precipitate previously seen are still present but in between there are now more finer intermetallic compound and phases. Prominently there are fine eutectic phase cluster in multiphase structure along with intermetallic compounds.

5.3 Sample 9: Al-4Cu-6Mg-4Zn-0.2Mn-4Ni-1Ti

5.3.1 Sample 9: Casted Alloy and Microstructure

Experimental Run 9	
	

Table 23: Images of resulting casting for Al- 4Cu- 6Mg- 4Zn- 0.2Mn- 4Ni- 1Ti.

Metal with deviation from orthogonal array weight.		
Metal	Stated weight	Actual weight
Cu	40	39
Ni	105.9	107

Table 24: Deviation of weight for experimental run 9.

Weight of experimental run 9 is 911.5 g which is of expected level due to alloy burnt and oxidising. One observation made is the lack of shrinkages in the casted alloy of experimental run 9 which can be attributed to the combination of constituents in the alloy.

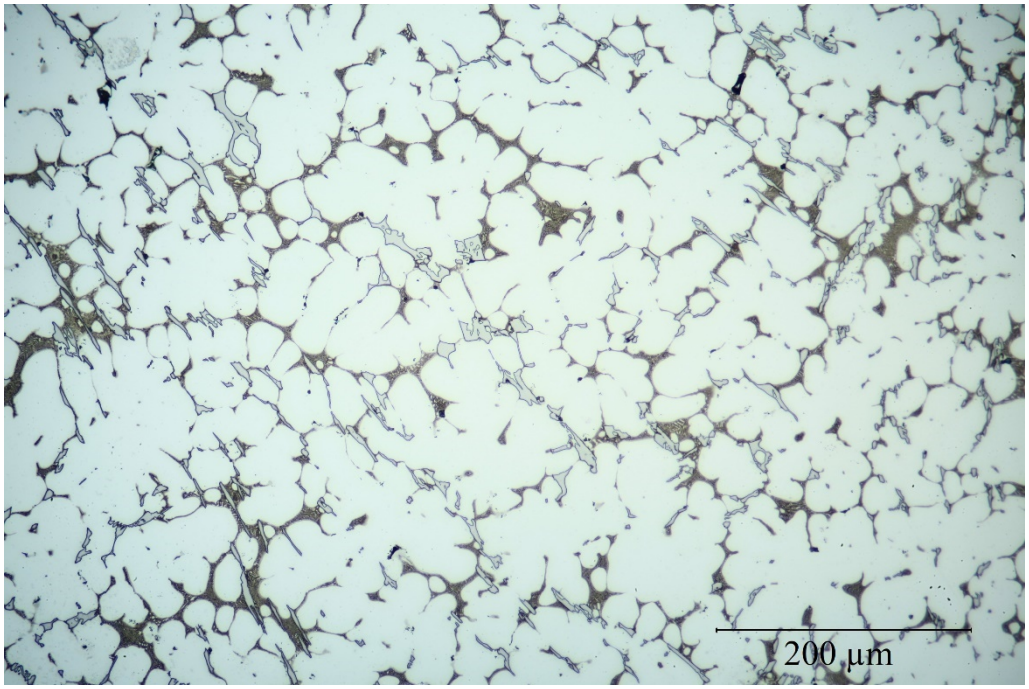


Figure 26: Microstructure of as- cast for Al- 4Cu- 6Mg- 4Zn- 0.2Mn- 4Ni- 1Ti.

Microstructure of sample 9 shows high amount of alpha phase aluminium phase. With significantly less secondary phase. Reduction in number of secondary phases can be linked to the lower amount of alloying element added leading to lower number of impurities and undissolved intermetallic compound. Referencing table 11, there are only 6 alloying elements compared to 9 alloying element of alloy 7 and 8. Eutectic phase shown in figure 24(B) for sample 9 is finer and in fibrous shape differing from sample 7 and 8 with another secondary intermetallic compound near or on it forming a multiphase structure, shown in figure 24(A). There are also no chinese script shape eutectic phase which is seen in sample 8. Finer eutectic phase and fewer intermetallic phase obtained can be also linked can be reasoned to the absence of chromium and silicon in alloying constituents. Chromium

and silicon has limited solid solubility in aluminium. Solubility of chromium, in particular, is very low where generally no more than 0.35% is added before coarse precipitate is generated. Consequently, silicon nucleates on aluminium phosphite particle in melt to form non-dissolvable impurities. [52] Observation can be corroborated by microstructure results obtained for sample 7 and 8 which has coarse intermetallic phase and chromium percentage over the solubility limit at 0.4%.

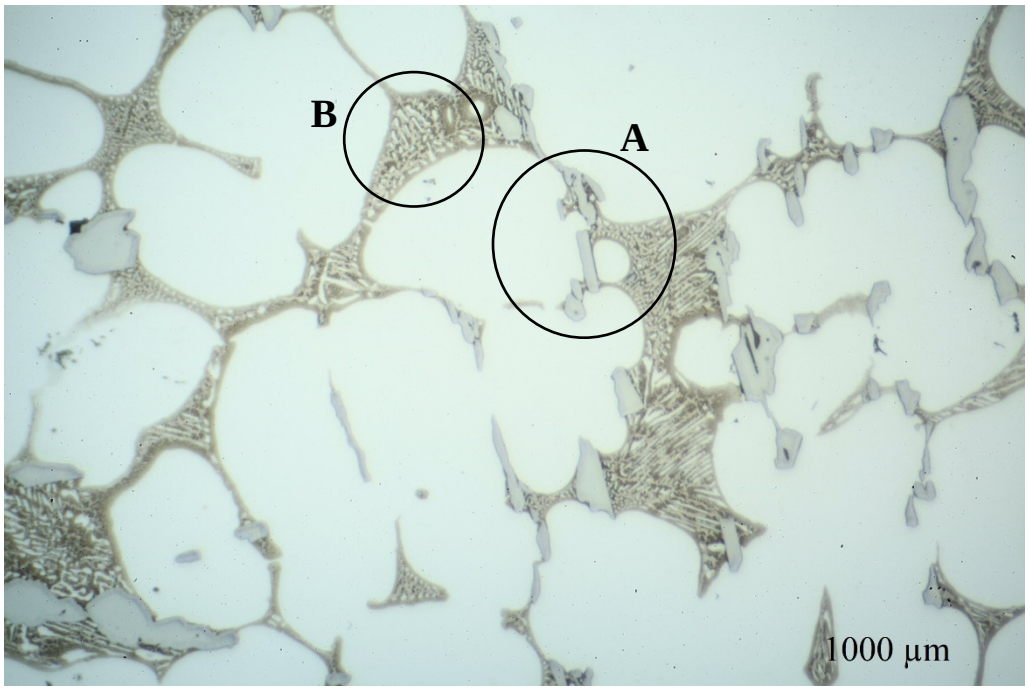


Figure 27: Alloy 9 as- cast microstructure at 500x magnification.

The undissolved intermetallic phase seen in the figure 19 is predominantly platelets shaped with a little needle shape intermetallic phase.

Sample 9: As- Cast Vickers Hardness (HV)				
132.7	136.7	130.7	138.2	125.4
Average Hardness: 132.7				

Table 25: Sample 9 average as- cast hardness.

The average as-cast hardness of alloy 7 shows intermediate hardness in comparison to common commercialised aluminium alloy of 1xxx to 7xxx series with hardness in the range of 30 to 160 HV. [62]

5.3.2 Sample 9: Solution Treatment Results

The optimum solution treatment for sample 9 is at temperature of 510 °C with 6 hours holding time.

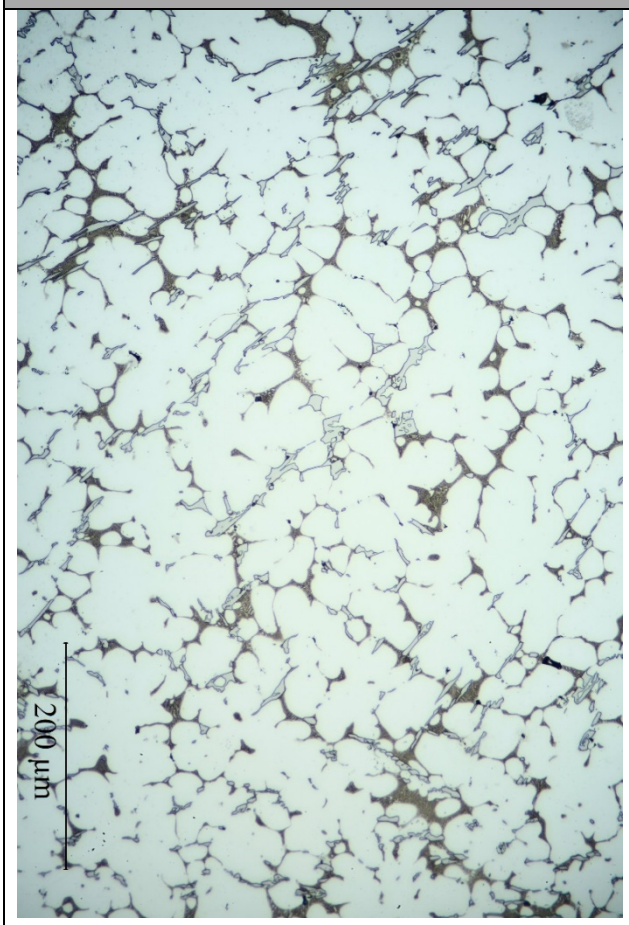
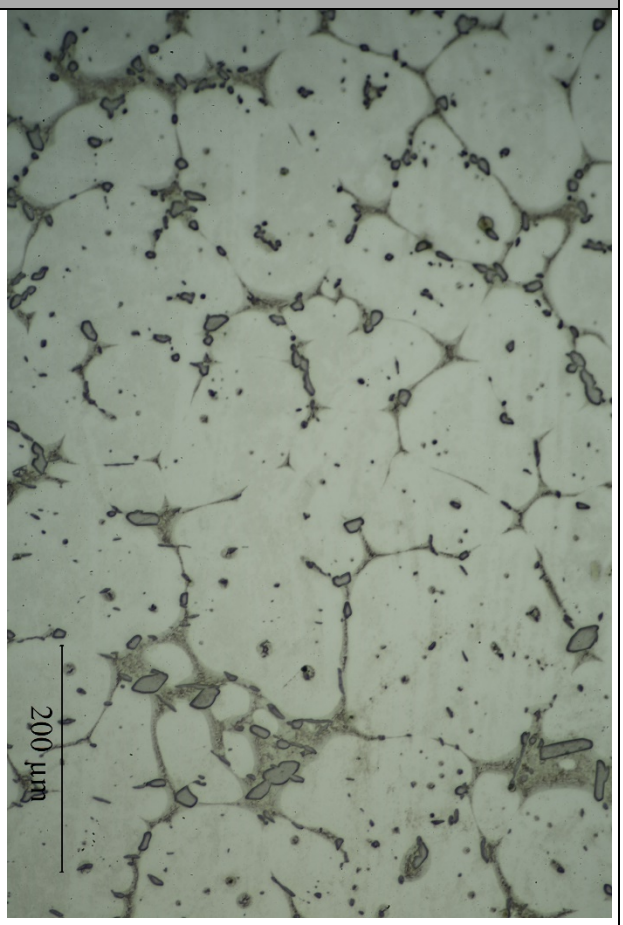
As- Cast Sample 9	Solution Treated Sample 9
	

Table 26: Compares sample 9's as-cast and solution treated microstructure.

From observing both as- cast and solution treated alloy 9 microstructure, it is clear that most intermetallic phase has dissolve or spherodised. The needle and platelet-shaped intermetallic has dissolved and coagulated into the sporadic intermetallic compound of varying shape and size seen in the solution treated sample. The multiphase structure is still present. Eutectic phase, on the other hand, has dissolved completely to the α-phase aluminium or has spherodised into fine intermetallic compound. Intermetallic compound does not exhibit coarsening process observed on alloy 7 and 8 after solution heat treatment. One possible explanation is the high percentage of Mg in alloy 9 which prevent clustering according to literature review.

5.3.3 Sample 9: Aging Curve and Aged Hardness Results

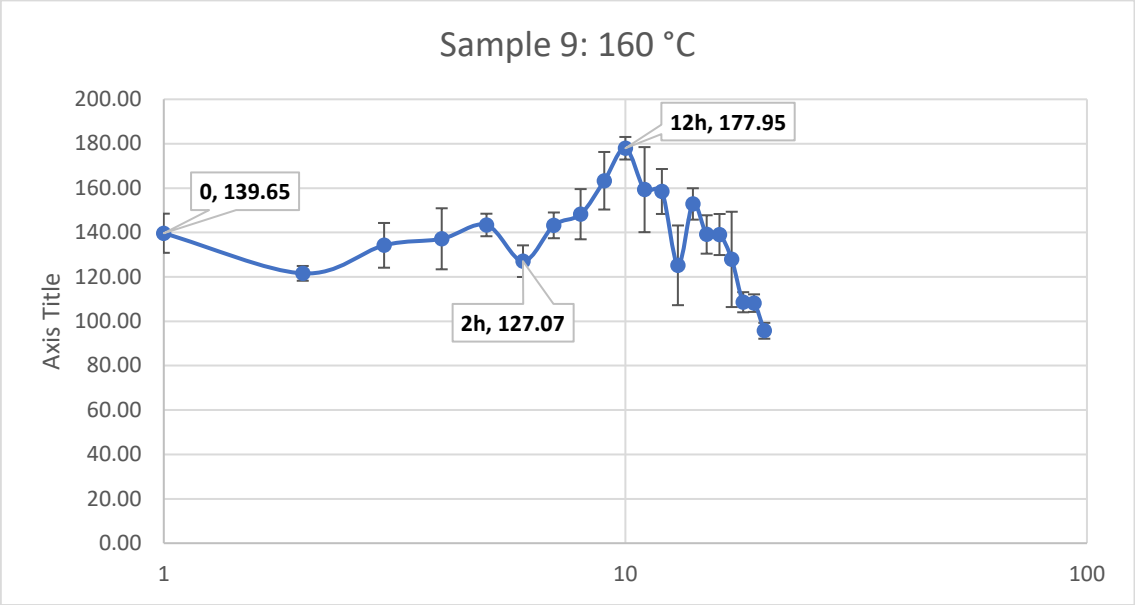


Figure 28: The plotted aging curve which yields peak hardness for alloy 9 after aging at 160 °C.

Sample 9 produces almost similar hardness to sample 7. Sample 9 has peak hardness of 177.95 after aging for 12 hours at temperature of 160 °C. In contrary to sample 7 though, the increment obtained from age hardening is the lesser though, with a 40 HV increment from the as-cast hardness of 132 HV. Hardness reading at 0 in figure 28 is solution treated hardness. Error bars on the aging curve signify the standard deviation of hardness readings. Hardness value obtained can be assumed to be consistent to an extent, as the error bar is quite large in figure 28 signifying large data range logged.

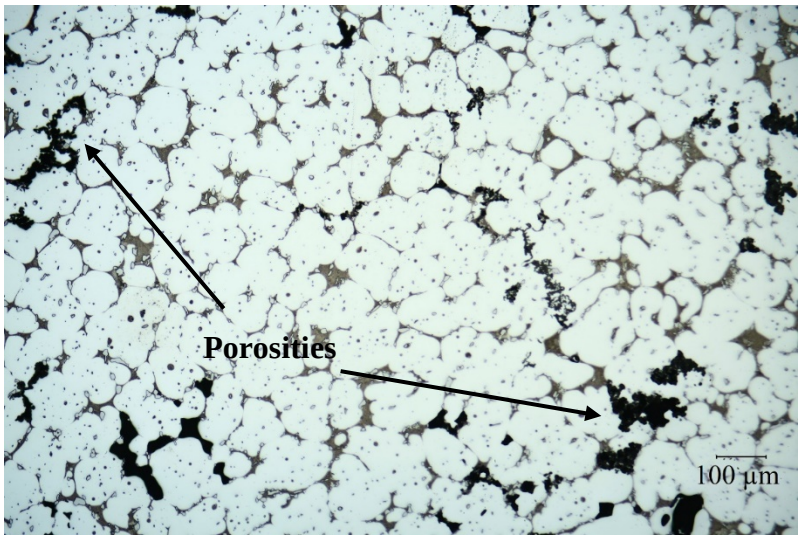


Figure 29: Porosities seen on sample 9 after heat treatment.

Sample 9 after completing solution heat treatment and age hardening also shows significant micro-sized porosities across the surface of alloys. These microporosities on certain age hardened sample 9, generate significant defects enough to cripple their hardness and strength. This shows on the aging curve which has more irregularities observed compared to sample 7 and 8. Overall sample 9 has below average respond to heat treatment compared to alloy 7 and 8 due to aforementioned issues.

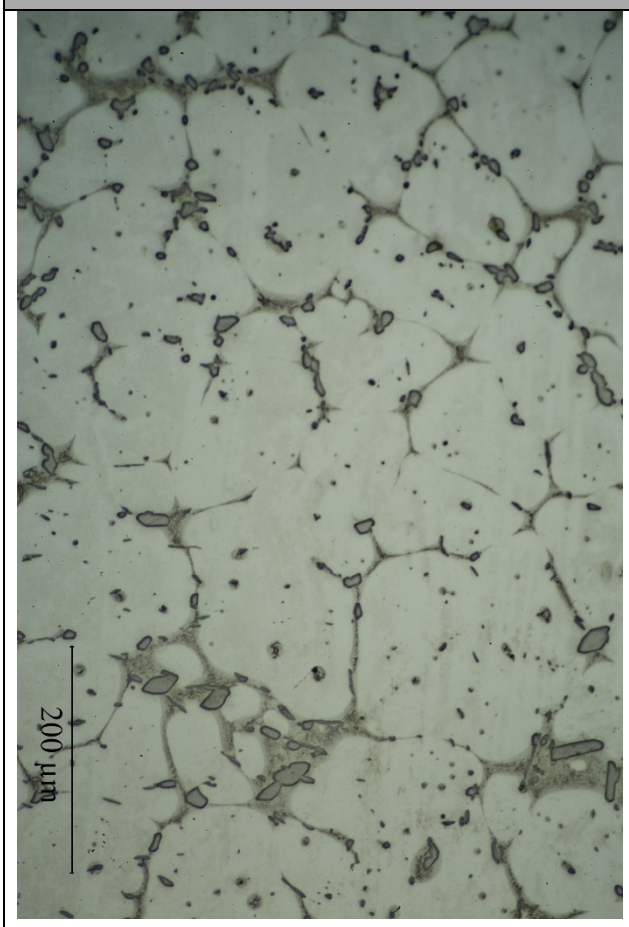
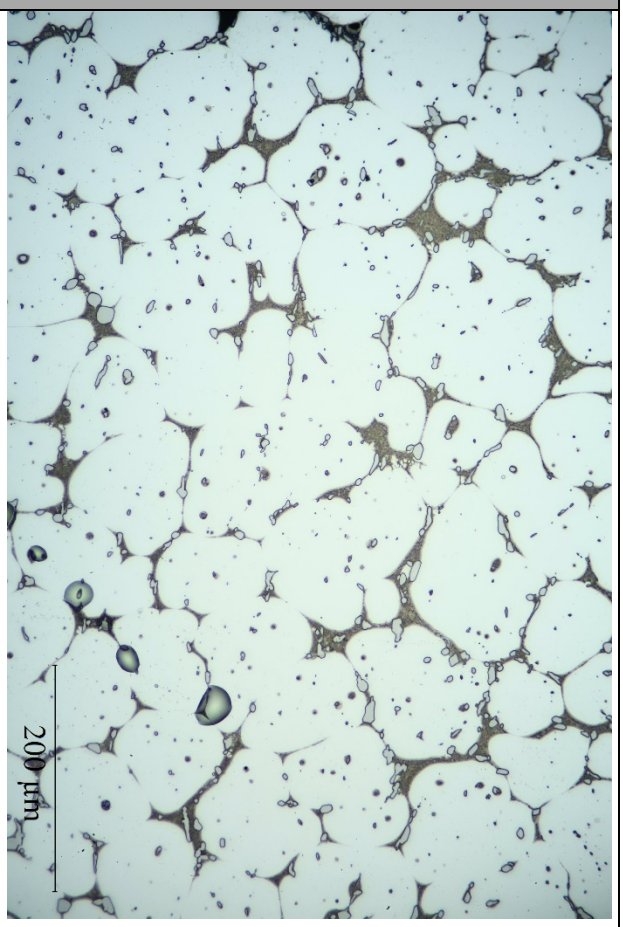
Solution Treated Sample 9	Peak Aging Microstructure Sample 9
	

Table 27: Compares the solution treated and after age hardening microstructure for sample 9.

The microstructure obtained after peak age hardening does not shows any significant difference with the solution treated microstructure. One small difference observed is the number of fine precipitates dispersed on the surface of alloy 9, compared to solution treated microstructure which has some bigger sized intermetallic particles. Observation is corroborated with the hardness results obtained where there is no significant increase in hardness as well. A theory for indifference observed after age hardening is the absence of chromium in alloy. According to table 1, chromium controls the grain structure and prevent recrystallisation of intermetallic compounds during heat

treatment. Without chromium in the alloying constituents, the previously undissolved intermetallic compound recrystallises back after age hardening instead of forming fine intermetallic precipitates.

5.4 Orthogonal Array Analysis Result

Using the peak hardness obtained for each experimental run, beneficial effect from varying the factor level or the alloying percentage to the hardness of aluminium alloy can be inferred. Through this analysis, optimum alloying percentage can be detected as well because higher percentage does not correlate to higher hardness according to literature review.

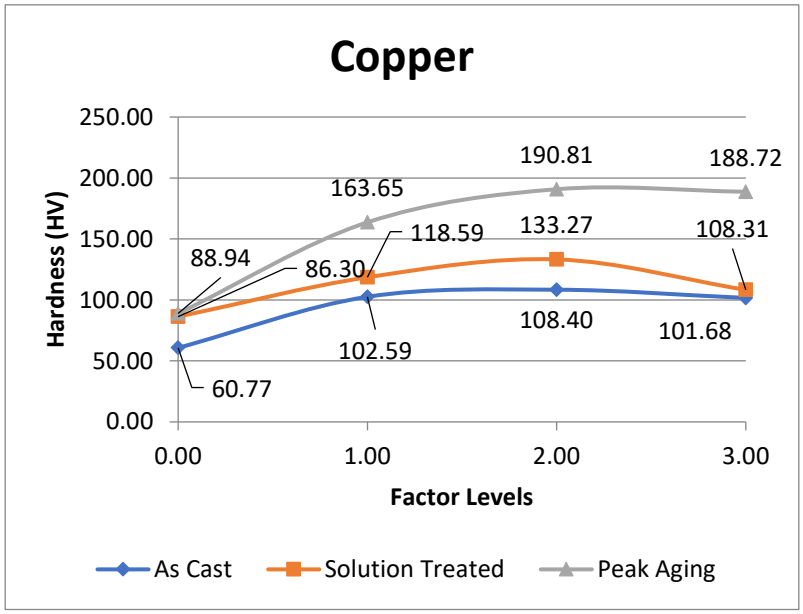


Figure 30: Copper influence on hardness of aluminium alloy.

The effect of on the hardness of aluminium alloy can be seen to increase up to factor level of 2, corresponding to alloying percentage of 4% and then equalise after with no rise in hardness observed for factor level of 3. Implying that addtion of copper beyond 4% will not further improve hardness or decrease hardness of aluminium alloy.

Result obtained is in line with literature review in section 3.1 pertaining to influence of copper on aluminium alloy hardness which max out at 4% to 6%.

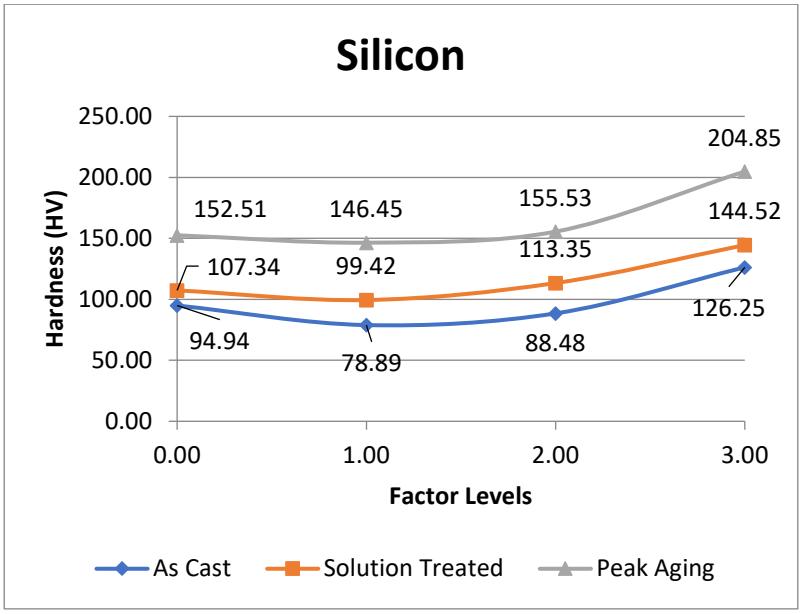


Figure 31: Silicon influence on hardness of aluminium alloy

Average hardness of alloy increases exponentially at factor level of 3 which corresponds to alloying percentage of 6%. Reasons pertaining to sudden increase is assumed to influences of other alloying constituents. Silicon tends to form intermetallic particles with other alloying elements such as magnesium to form $MgSi_2$, manganese to form spherodise particles that is finer and more. The rise in hardness is documented by Vipin Kumar et al. noting the rise of hardness along with number of silicon particles but in expense of strength which will decrease and formation of coarser silicon particles. [65]

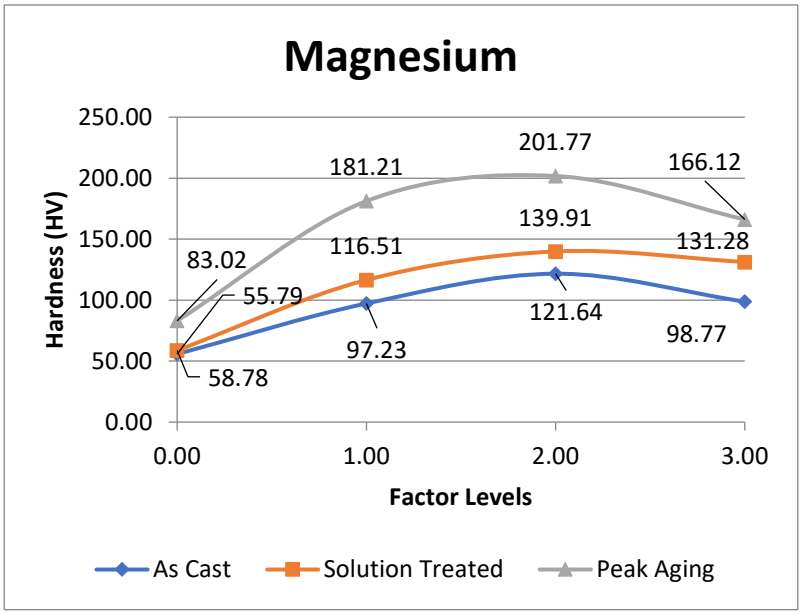


Figure 32: Magnesium influence on hardness of aluminium alloy.

Magnesium increases the average hardness of aluminium alloy up to factor level of 2 before deteriorating the hardness of aluminium alloy after. Implying that alloying percentage beyond 4% corresponding to factor level of 2 is the alloying limit for magnesium in aluminium alloy. Current commercialise aluminium alloy does not have magnesium content in excess of 5.5% also. Alloying aluminium with magnesium in excess of 5.5% produces alloy susceptible to intergranular cracking and stress corrosion because magnesium will precipitate preferentially at a high anodic phase. [66]

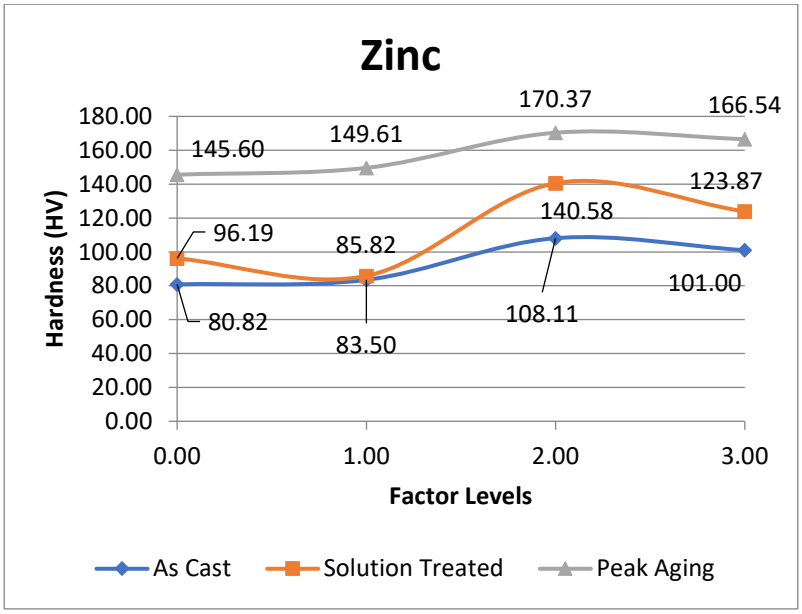


Figure 33:Zinc influence on hardness of aluminium alloy.

From figure 33, average hardness of alloy increase as alloying percentage of zinc increases. These trends max out at factor level of 2 which corresponds to alloying percentage of 6%. Zinc is susceptible to induce stress corrosion cracking that increases with proportion of zinc percentage in aluminium alloy.

Susceptibility of zinc to the stress corrosion cracking is exacerbated when there is presence of magnesium in the alloying composition as well. Stress corrosion cracking is a defect that reduces the mechanical properties of alloy.

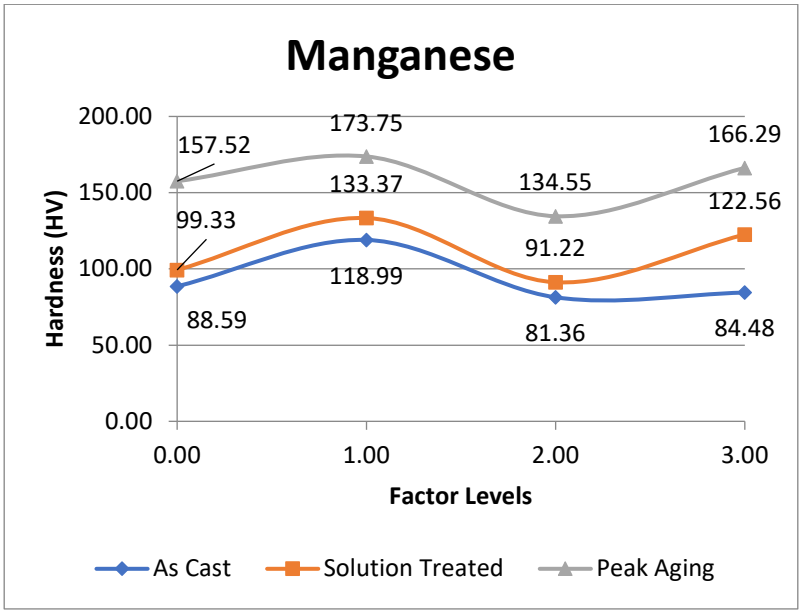


Figure 34: Manganese influence on hardness of aluminium alloy.

Influence of manganese on average hardness of aluminium alloy seems to be irregular whereby a rise in hardness is observed when factor level 1 is applied but decreases again at factor level 2 to finally increase again at factor level of 3. Reason for dip in hardness at factor level 2 can't be ascertained.

Result ontained is deemed irregular because manganese is added to control the grain structure and form hard intermetallic phase with zinc, magnesium and copper. Increasing the alloying percentage of manganese should lead to finer grains elevating the overall hardness of alloy samples unless

certain range of alloying percentage induce the formation of as of yet undiscovered intermetallic compound that causes low hardness in alloy. An experiment by Soo Woo Lee and Duck Hee Lee back the claims made as they found that manganese added to aluminium alloy at range of 0.7% to 1.2% increases the mechanical properties of aluminium alloy possessing zinc and magnesium as well. Although, alloying constituents between the ones used by Soo Woo Lee and Duck Hee Lee and the ones casted in this thesis is not same, the journal proves that hardness at higher manganese percentage is possible. [67]

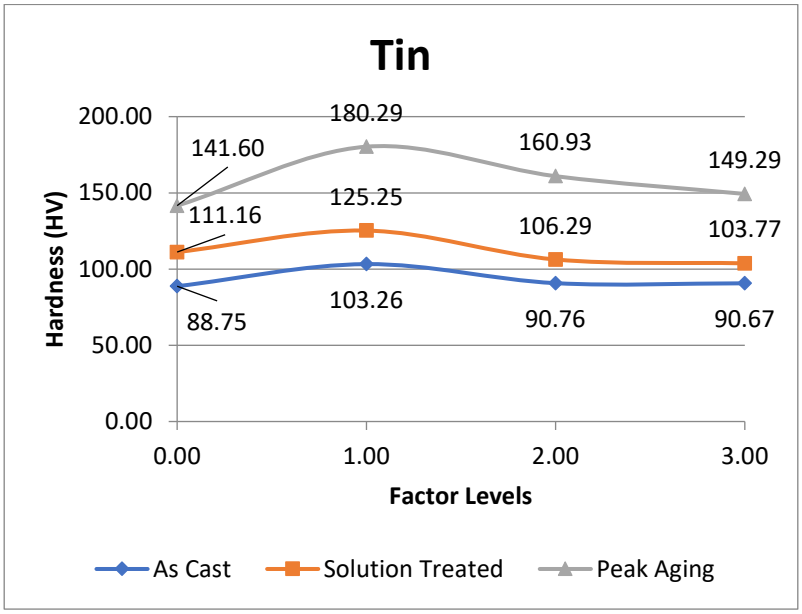


Figure 35: Tin influence on hardness of aluminium alloy.

The plot obtained in figure 35 exhibits a trend where hardness increases proportionally until factor level of 1 and decreases steadily afterwards. Tin can only be added in small percentage. Results obtained shows that alloying limit for tin is between 1% to 2% of aluminium alloy with these alloying constituents.

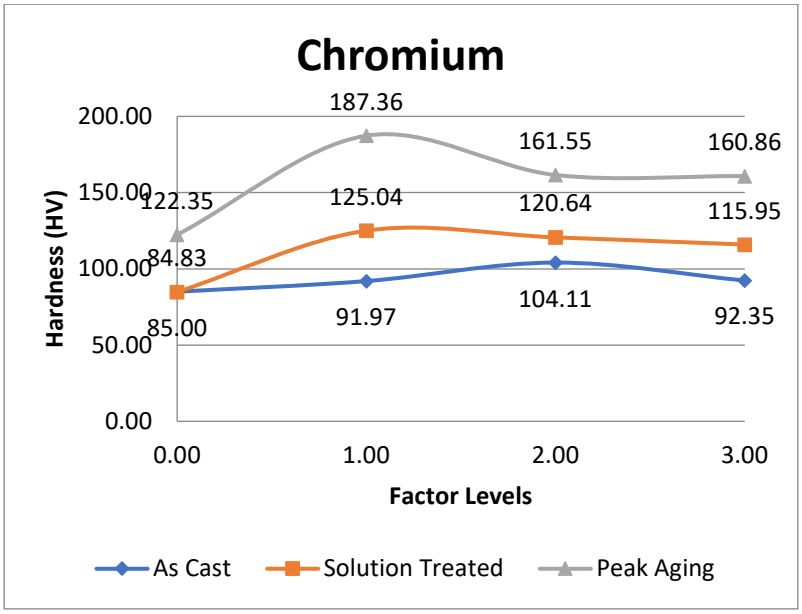
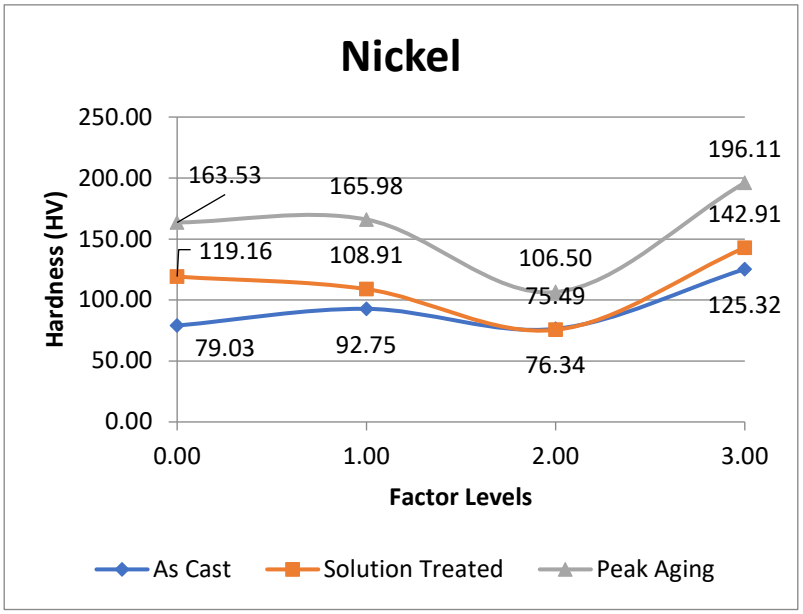


Figure 36: Chromium influences on hardness of aluminium alloy.

Chromium influence on hardness of aluminium alloy is simular to hardness trend exhibited by tin. The average hardness peaks out at factor level of 2 before steadily decreasing after. Chromium in excess of 0.35% tends to form coarse constituents with impurities in the aluminium alloy such as manganese and titanium in this case. This explains for the downward trend obtained after factor level of 2 which corresponds with chromium alloying percentage of 0.4% in aluminium alloy.



. *Figure 37: Nickle influence on hardness of aluminium alloy.*

The average hardness for each alloying elements shows irregularities in the trend as well. Hardness level was steady at about 160 HV at factor level 0 and 1 before dropping at factor level of 2 and then rising exponentially at factor level of 3. Nickel generally is able to raise the hardness of aluminium up to 2%. At factor level of 2, hardness should increase as it corresponds to alloying percentage of 2% nickel. Hence the irregularities as not only does hardness decrease instead but hardness increases again at factor level 3(3%) of aluminium alloy.

6. DISCUSSION

Previously in section 5, the term ‘undissolved intermetallic compound’ or words to that effect was quoted heavily while describing the microstructure of the alloys. Addition of so many alloying constituents and respective percentage will exceed the solubility limit of aluminium. Another possible cause of microstructural inhomogeneity is liquid segregation phenomena during casting.

Sample 8 has highest hardness in relation to other samples can be related to the high concentration of fine precipitation which has formed. Dislocation movement is impeded in proportion to higher amount of fine grains according to literature review, leading to higher hardness. This correlates with the results obtained as well where peak hardness of alloy 8 is much higher than that of alloy 7 and 9 which has less or no fine precipitate after age hardening.

The aging curve plotted in figure 19, 24 and 28 does not exhibit a smooth trendlines. Few aged samples show an unreliable reading of lower or higher hardness which is not in line with the trends of aging. An example will be the 2-hour aged sample for sample 9 in figure 28 which is not in conformation with the trend of the plot. Aging of alloy should ideally follow a smooth plotlines with 3 identifiable regions of underaging(rising trend), peak aging(peak) and overaging(decreasing trend) similar to figure 16. In reality, these smooth plotlines are hard to attain due to defects and alloying composition which is not even after casting. It is common issues to encounter these issues on aluminium alloy which is cast with conventional gravity casting which has non-equilibrium cooling and turbulent pouring into mould cavity promotes segregation during casting. When heat treated the compromised area of casting will produce abnormally high or low mechanical properties after heat treatment.

Based on the results obtained from orthogonal analysis, figure 30 to 37, optimum alloying constituents has been identified. The ideal alloying composition of aluminium alloy based on analysis of orthogonal array are:

1) Silicon: at or increased beyond 6% indefinitely until detrimental properties is obtained as the figure 31 shows the hardness increasing in proportion with factor level.
2) Copper: 4% to 6%. Average hardness tops out at factor level 2 and 3 as seen in figure 30. Further increase in copper percentage will be futile as average hardness values does not show incremental rise from factor level 2 to 3 and holds steady.
3) Magnesium: Based on figure 32, the peak hardness of magnesium is at factor level 2. So alloying percentage should be 4%.

4) Zinc: Similar to copper the zinc percentage in aluminium should be kept at 4% to 6%. Further increase will be futile as peak hardness is achieved and holds steady.
5) Manganese: It is clear from analysis hardness peaks at factor level of 1. Thus, for following casting the percentage of manganese should be 0.2%.
6) Tin: Peak hardness is obtained at factor level of 1 according to figure 35. Ideal alloying percentage of tin for the next casting is 1%.
7) Nickel: The peak average hardness of nickel according to orthogonal array analysis is at 3% or factor level 3. For following iteration, alloying percentage beyond 3% can be tested to see if there is further increment in hardness obtained.
8) Chromium: Hardness of chromium clearly peak out at factor level of 1 which correspond to 0.2%.

Further analysis of results is done to validate and relate the number of alloying elements to hardness of aluminium alloy. Table 10 from section 4.2 is revised with ideal alloying percentages identified highlighted.

	Factor levels				
Elements	**0**	**1**	**2**	**3**	
Si	0	2	4	6	wt%
Cu	0	2	4	6	wt%
Mg	0	2	4	6	wt%
Zn	0	2	4	6	wt%
Mn	0	0.2	0.4	0.6	wt%
Sn	0	1	2	3	wt%
Cr	0	0.2	0.4	0.6	wt%
Ni	0	1	2	3	wt%

Table 28: Revised factor level table with optimum alloying percentage identified.

In relation to the ideal optimum identified for each alloying elements, orthogonal array table in section 4.2 can be used to validate if optimum alloying percentage stated produces alloys with high hardness.

Experiment Runs	Si	Cu	Mg	Zn	Mn	Sn	Cr	Ni	Peak Aging Hardness	Total Factor level	No. of optimum composition
1	0	0	0	0	0	0	2	2	34.68	4	0
2	1	0	0	3	2	3	0	2	57.56	11	1
3	2	0	3	2	3	1	3	0	149.58	14	2
4	3	3	2	0	0	1	1	0	193.42	10	5
5	0	3	2	0	3	3	2	3	192.54	16	3
6	1	3	1	3	0	3	3	1	197.1	15	2
7	2	3	1	1	3	1	0	2	171.82	13	2
8	3	2	2	3	1	0	2	3	240.25	16	6
9	0	2	3	2	1	0	0	2	177.95	10	3
10	1	2	1	3	3	0	1	0	185.13	11	3
11	2	0	3	0	2	3	1	1	163.87	12	1
12	3	1	2	2	1	2	2	3	180.87	16	5
13	0	1	3	2	0	2	1	1	211.9	10	2
14	1	2	0	1	2	2	3	0	168.4	11	1
15	2	1	1	1	2	0	3	3	181.3	13	1
16	3	1	0	1	1	2	0	1	103.73	9	2

Table 29: Revised orthogonal array table with peak aging hardness and optimum composition highlighted.

The yellow boxes signify optimum alloying percentage and red box signify the alloys which contribute the most to hardness properties according to orthogonal array results. Studying table 29, a conjecture can be made that hardness of alloy is indeed not dependant on the alloying percentage. Dependency on alloying percentage is rule out as some alloy with low total factor level equivalent to the alloying percentage has higher hardness compared to alloys with high total factor level. For example, alloy 4 and alloy 3. Higher percentage of alloy does not contribute to higher hardness. Bigger factor affecting hardness properties is alloying the aluminium with respective alloying element's optimum percentage and ensure alloying elements which contribute to higher average hardness are included in the constituents of aluminium alloy. Statement before is proven as alloy 8 has highest peak aging hardness among all 16 alloys as well as highest number of optimum alloys in the constituents with high percentage of silicon, nickel, copper and magnesium in composition.

Furthermore all alloys samples with at least 3 of 4 between silicon, nickel, copper and magnesium produces alloy with at least 170 HV in hardness which is above average commercial aluminium alloy hardness at 140 HV.

6.1 Future Research

Designing an alloy according to literature research done is not a onetime only process. When promising alloy is found by initial research, repeat of the same general process beforehand with better optimisation and control is required to obtain applicable alloy. Based on results and discussion for alloy 7, 8 and 9, there are clear improvements to the made for future iteration of the experiment. While objectives of the thesis to identify new aluminium alloy with higher than has been achieved, more work needs to be done for the findings to have any practical world use. For real world application, final product after casting should not have any porosities and defects which are found in most samples.

First, the casting process has to be better controlled. Current casting methods produces alloy with porosities in some samples such as sample 8, alloy segregation and shrinkages. One solution suggested was increasing the casting temperature to increase the casting fluidity. A casting temperature of 780 °C was used for casting process previously, a further increment of 50 °C was suggested to be sufficient. Gravity filling operations encourage turbulent behaviour of liquid metal producing substantial entrainment of oxides films. Trapped in liquid metal, the oxide films generate micro cracks(sample 9) and casting defects(sample 8). [68] Turbulence behaviour is the inconsistent speed and direction where liquid metal travels through the casting. A method to introduce a constant rate of pouring needs to be investigated and implemented as too fast pouring will cause turbulence while slower pouring rate cause execration of non-equilibrium cooling. [69] Shrinkages is a common problem and can be easily solve by the use of risers that contains and feeds additional molten metal to cavity during cooling. For commercialisation, automised casting process can be used.

The heat treatment process can be further optimised to improve homogenisation preventing the distortion and partial melting of alloy sample. A more controlled approach to solution heat treatment will be to hold the alloy at alloy a lower temperature for up to a day to partially homogenise the phases and then increasing to solution treatment temperature for a shorter period of time according to B. Geetha et al. [70] It is suggest for following iteration lower holding temperature can be set to about 30 °C lower than optimised solution treatment temperature and

held for a 12-hour. This lower the chances of distortion in aluminium alloy as longer holding time at near eutectic temperature will increase the chances of distortions.

Alloying constituents of following iteration shall follow the recommended constituents stated in discussion before. Few iterations should be done while varying the percentage of silicon and nickel beyond 6% while fixing the percentage of other constituents to percentage stated above. Silicon, copper, magnesium and nickel which produces alloy with higher average hardness should be tested further to obtain higher hardness.

7. CONCLUSIONS

Objectives set out at beginning of the thesis has partially achieved. A new composition of aluminium alloy was designed and tested out. Results shows that alloy 8 has peak hardness of 240 HV which is higher than commercial aluminium alloy hardness of 140 HV. Other sample documented on this thesis produce a hardness of 171 HV for sample 7 and 177 HV for sample 9. Modelling of alloy was designed by orthogonal array with alloying elements and constituents varied. Use of orthogonal array was able to successfully determine the effect of alloying element on hardness of aluminium alloy. 4 alloying elements was singled out for being able to produce alloy with higher hardness after orthogonal analysis which is silicon, nickel, magnesium and copper. Optimum alloying elements and percentage of addition for future iteration and research was also identified. Heat treatment on alloy samples was carried out successfully with alloys sample generally showing good response to heat treatment. Multiple trials and error were performed to successfully identify the optimum solution heat treatment temperature. The final heat treatment for sample 7 is at 540 °C with 3-hour holding time, sample 8 is at 540 °C with 3-hour holding time as well and lastly sample 9 at 510 °C and 6 hours holding time. Age hardening was done over a time period of 6 days at temperature of 160 °C, 180 °C, 200 °C and 230 °C. Sample 7 gain peak hardness at temperature of 200 °C with aging time of 1 hour 30 minutes. Sample 8 gain peak hardness at 200 °C and 30 minutes of aging time. Lastly, sample 9 obtain peak hardness aging for 12 hours at 160 °C. After aging, hardness of alloy at each time interval is logged and aging curve is plotted. Most sample is able to form fine precipitate after aging especially sample 8 after analysis of microstructure. Aging curve shows some outliers in results which can be reasoned to segregation of alloy composition and non-equilibrium cooling. Microstructure analysis is carried out on as-cast, solution treatment and aged sample. Degree of homogenisation observed using microstructure analysis is used to optimise solution treatment process. Sample 7, 8 and 9 shows

high number of intermetallic compounds and eutectic phase due to solubility limit of alloying elements in dominant aluminium α-phase as-cast conditions.

Overall, while results have been favourable, more work needs to be done before alloys designed can find applicability in the real world and be commercialise. Samples shows defects which is not acceptable for application, prominently the porosities seen in some samples. It is recommended that follow up research is done based on the findings of this thesis to better control and improve the processes used in this thesis to get better casting. Subsequent casting should be based on the recommended optimum alloying percentages instead of orthogonal array.

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9. APPENDIX

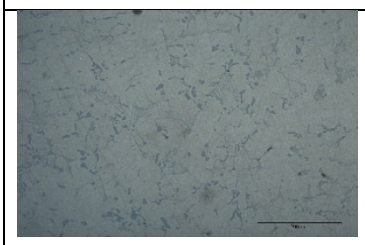
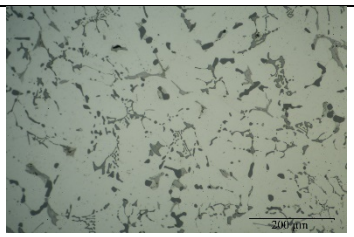
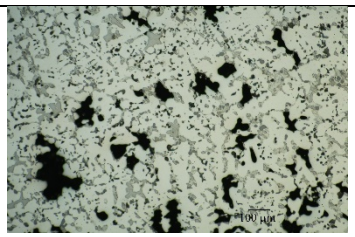
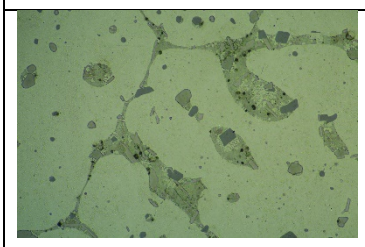
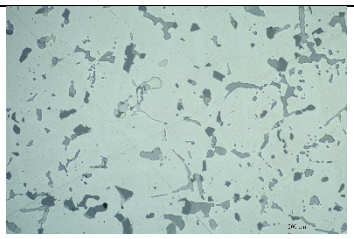
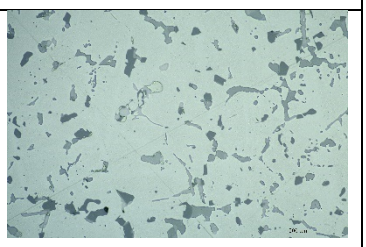
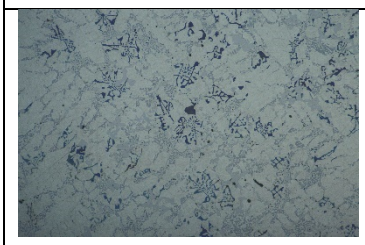
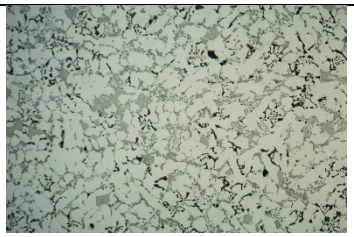
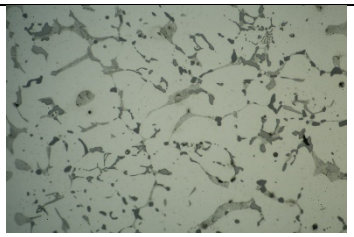
2 Hours 520 °C	2 Hours 530 °C	2 Hours 550 °C	2 Hours 570 °C
			Partially melted
Not enough homogenisation.	Not enough homogenisation.	Partial melting of some intermetallic compounds.	
12 Hours 540 °C	12 Hours 520 °C	3 Hours 540 °C(optimum)	
			
Distortion of sample.	Good sample homogenisation.	Same homogenisation as 12 hours but achieved with shorter time.	

Table 30: All the solution treatment iteration for sample 7.

2 Hours 520 °C	2 Hours 530 °C	2 Hours 550 °C	2 Hours 570 °C
			Partially melted
Not enough sample homogenisation.	Not enough sample homogenisation.	Mostly homogenise.	

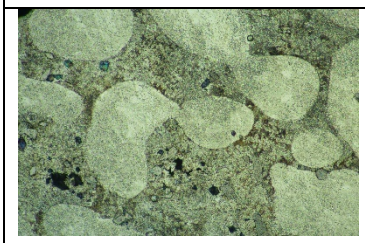
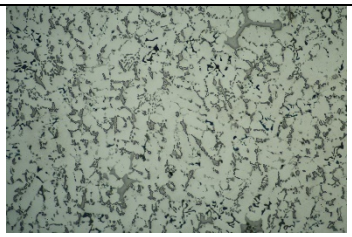
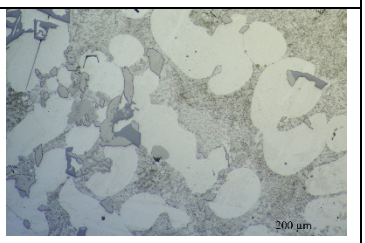
12 Hours 540 °C	6 Hours 510 °C	3 Hours 540 °C(optimum)	
			
Distortion and porosities.	Not enough homogenisation	Sample dissolve to fine particles.	

Table 31: All the solution treatment iteration for sample 8.

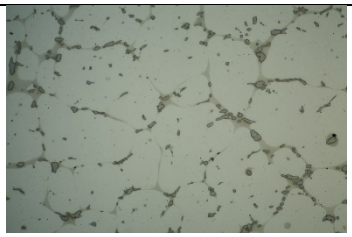
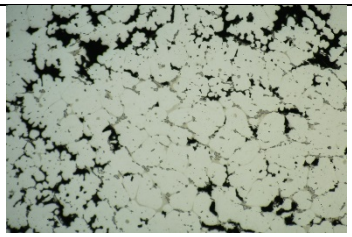
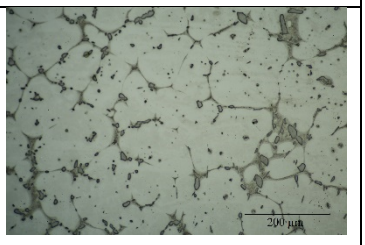
2 Hours 520 °C	2 Hours 530 °C	2 Hours 550 °C	2 Hours 570 °C
			Partially melted
Phases homogenises with few particles left.	Partial melting of phases.	High melting of phases.	
12 Hours 540 °C	6 Hours 520 °C	6 Hours 510 °C(optimum)	
Partially melted	Partially melted		
		Sample homogenise without partial melting.	

Table 32: All solution treatment iteration for sample 9.

Experiment Runs	Si	Cu	Mg	Zn	Mn	Sn	Cr	Ni	
1	0	0	0	0	0	0	2	2	E Chin
2	1	0	0	3	2	3	0	2	
3	2	0	3	2	3	1	3	0	
4	3	3	2	0	0	1	1	0	Jun
5	0	3	2	0	3	3	2	3	
6	1	3	1	3	0	3	3	1	
7	2	3	1	1	3	1	0	2	Victor
8	3	2	2	3	1	1	2	3	
9	0	2	3	2	1	0	0	2	
10	1	2	1	3	3	0	1	0	Lim
11	2	0	3	0	2	3	1	1	
12	3	1	2	2	1	2	2	3	
13	0	1	3	2	0	2	1	1	Ziwei
14	1	2	0	1	2	2	3	0	
15	2	1	1	1	2	0	3	3	
16	3	1	0	1	1	2	0	1	

Figure 38: Full factor table for all experimental run.

Calculation of composition in grams out of 1000g aluminium alloy									Note that 0.1wt% Al-3Ti-B		
Experiment Runs	Al-50Si(g)	Pure Cu(g)	Pure Mg(g)	Pure Zn(g)	Al-60Mn(g)	PureSn(g)	Al-10Cr(g)	Al-19Ni(g)	Al-3Ti-B(g)	Remaining Aluminium(g)	
1	0	0	0	0	0	0	40	105.26	1	853.7368421	E Chin
2	40	0	0	60	6.67	30	0	105.26	1	757.0701754	
3	80	0	60	40	10	10	60	0	1	739	
4	120	60	40	0	0	10	20	0	1	749	Jun
5	0	60	40	0	10	30	40	157.89	1	661.1052632	
6	40	60	20	60	0	30	60	52.63	1	676.3684211	
7	80	60	20	20	10	10	0	105.26	1	693.7368421	Zach
8	120	40	40	60	3.33	10	40	157.89	1	527.78	
9	0	40	60	40	3.33	0	0	105.26	1	750.4068421	
10	40	40	20	60	10	0	20	0	1	809	Victor
11	80	0	60	0	6.67	30	20	52.63	1	749.6984211	
12	120	20	40	40	3.33	20	40	157.89	1	557.7752632	
13	0	20	60	40	0	20	20	52.63	1	786.3684211	Ziwei
14	40	40	0	20	6.67	20	60	0.00	1	812.33	
15	80	20	20	20	6.67	0	60	157.89	1	634.4352632	
16	120	20	0	20	3.33	20	0	52.63	1	763.0384211	

Figure 39: Full composition of alloys in gram for all experimental runs.

Sample 7	as- cast	83.8																			
160 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	78.4	77.5	107.3	123.7	133.8	146.7	144.3	141	142.9	152.3	153.7	156.4	156.1	149.1	118.9	117.1	103.8	114.6	114.2	94
	2	72.5	82.1	103.2	127	130.6	146.4	142.5	155.9	140.9	167.7	165.1	147.4	137.6	142.4	124.5	120.9	109.4	77.8	113.2	102
	3	71	85.4	97.2	130.5	127.4	147.1	146.7	145.7	153.5	153.2	148.6	140.2	140.6	135.2	118.4	133.3	103.2	121.8	118.8	112.2
	4	77	77.6	100.5	129.7	137.4	141.8	136.5	157.6	135.5	156.2	170	139.3	138.6	153.1	103.4	133.2	97.3	83.9	121.6	109.1
	5	68.4	73.5	98.3	124	133.2	136.5	130.3	148.9	152.5	157.6	152.3	148.7	157.8	155.3	103.2	132.3	89.5	120	124.4	116.1
Mean Hardness		73.46	79.22	101.30	126.98	132.48	143.70	140.06	149.82	145.06	157.40	157.94	146.40	146.14	147.02	113.68	127.36	100.64	103.62	118.44	106.68
Standard Dev		4.17	4.60	4.06	3.14	3.74	4.56	6.63	6.95	7.75	6.15	9.13	6.98	9.95	8.23	9.77	7.76	7.56	21.06	4.77	8.77
180 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	78.4	90.7	87.4	98.3	111.5	148.4	139.4	133.2	136.5	136.1	138.1	155.7	152.3	151.1	125.3	116.1	118.3	125.3	95.4	111.2
	2	72.5	86.5	81.9	97.2	111.4	120	138.8	141.1	134.7	141	155.5	142	166.9	139.8	132.7	126.8	114.2	114.4	113.8	102
	3	71	89.6	81	101.9	115.7	125.4	129.2	132.3	139.6	136.1	158.4	153.2	142.7	125.2	129.5	127	111.5	104.7	102	115.1
	4	77	81.1	88.9	108.2	105.4	142.6	136.2	141.6	138.3	154.4	137	160	145.8	139.1		120.4	126.6	120	102.3	105.6
	5	68.4	101.7	86.6	92.7	98.7	134	147.1	154.7	149.6	145.3	144	156.8	136.3	134.3		112	105.4	109.7	93	106.5
Mean Hardness		73.46	89.92	85.16	99.66	108.54	134.08	138.14	140.58	139.74	142.58	146.60	153.54	148.80	137.90	129.17	120.46	115.20	114.82	101.30	108.08
Standard Dev		4.17	7.57	3.50	5.79	6.61	11.74	6.44	9.00	5.81	7.64	9.87	6.90	11.64	9.40	3.71	6.59	7.91	8.15	8.08	5.11
200 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	78.4	145.1	142.6	148.1	171.5	156.4	165.3	160.7	159.6	131.6	150.1	150.6	141.4	125.9	119.8	122.6	109.7	114.5	96.3	89.7
	2	72.5	150.7	146.3	150.2	174.9	156.3	163.6	163.1	162.3	165.3	155.1	150.1	144.9	138.7	125.1	105.6	102.4	117	97.3	94.2
	3	71	137.2	156	157.2	172.2	156.9	168.1	152.4	147.2	165.3	143.7	148.4	138.11	151.9	112.4	121.3	111.7	115.3	89.5	92.9
	4	77	139.2	147	156.4	173.9	188.6	162.6	169.3	156.2	160.6	151.2	145.1	154.3	159.3	114.2	121.9	103.7	124.4		92
	5	68.4	142.7		156	166.6			159.6	161.8	162.3		161.5	130.6	120.8	135	122	107.7	113.6		87.1
Mean Hardness		73.46	142.98	147.98	153.58	171.82	164.55	164.90	161.02	157.42	157.02	150.03	151.14	141.86	139.32	121.30	118.68	107.04	116.96	94.37	91.18
Standard Dev		4.17	5.29	5.69	4.13	3.21	16.04	2.41	6.11	6.20	14.35	4.73	6.18	8.73	16.44	9.14	7.33	3.93	4.34	4.24	2.81
230 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	78.4	108.8	94.2	133.9	127.9	135.1	136	120.1	116.4	104.8	90.2	94.9	102	98	89.5	84.6	92.3	79.5	81.6	80.8
	2	72.5	114.4	118.2	138	136	123.2	117.2	109.7	108	96.6	98.4	90.9	91.7	92.9	85.5	78.4	83	76.1	84.3	74.6
	3	71	127.8	118.3	116.7	125.7	126.1	138.3	121.9	94.1	88.4	93.5	92.4	100.8	88.6	90.9	74.2	79.8	82.6	80.1	82.7
	4	77	114.87	128.2	112	121.2	125.6	119.7	110.9		100.5	99.1	96.2	104.8	93.7	87.1	83.7	80.1	84.3	76.9	82.2
	5	68.4	116.8		116.9													80.3	80.3		60
Mean Hardness		73.46	116.53	114.73	123.50	127.70	127.50	127.80	115.65	106.17	97.58	95.30	93.60	99.83	93.30	88.25	80.23	83.10	80.56	80.73	76.06
Standard Dev		4.17	6.97	14.46	11.62	6.20	5.22	10.89	6.24	11.26	6.97	4.22	2.39	5.67	3.85	2.41	4.86	5.30	3.13	3.09	9.54

Table 33: Age hardened sample 7 hardness log.

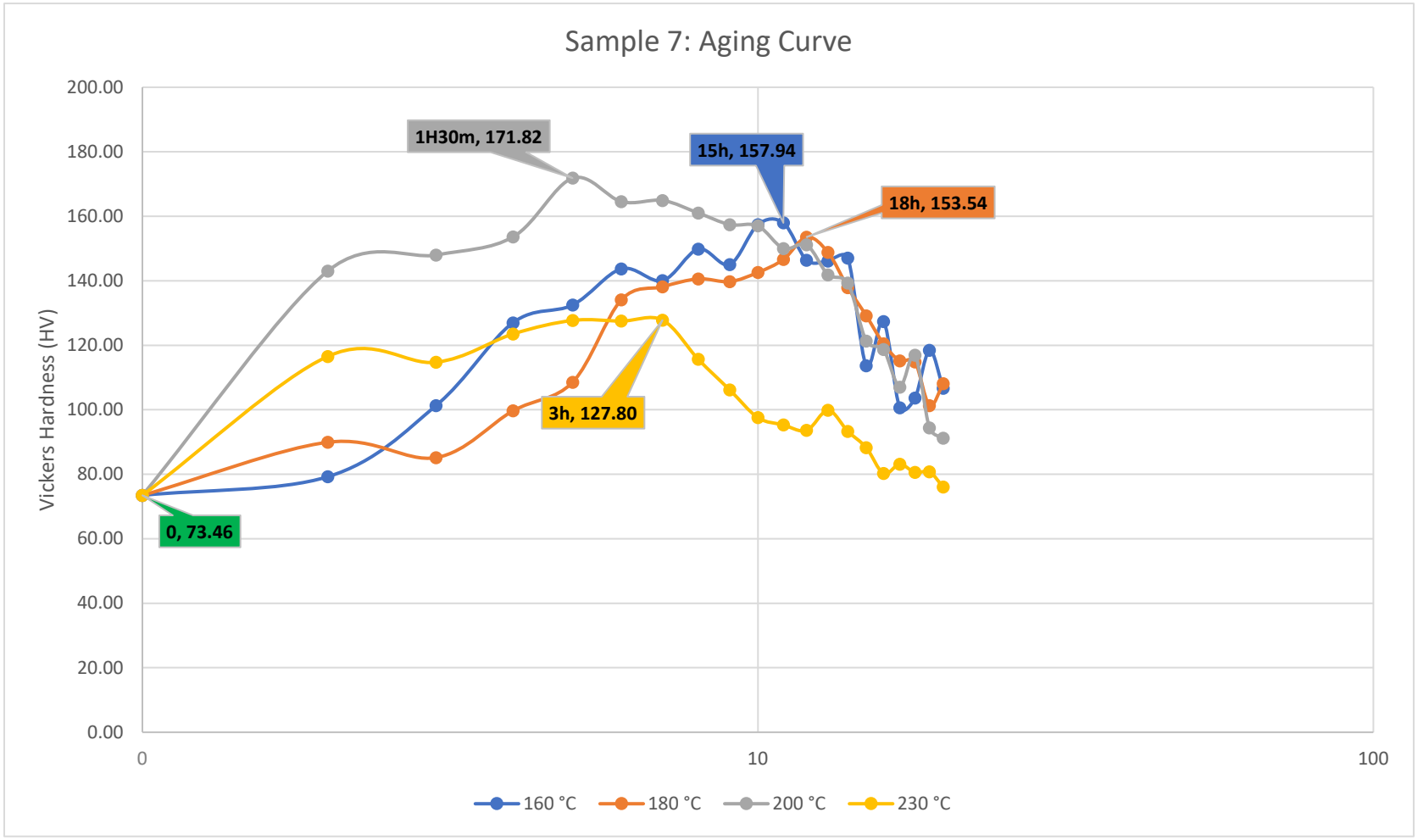


Figure 40: All plotted aging curve for sample 7.

Sample 8	sample 8	as- cast	157																		
160 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	167.1	179.7	189.9	173	193.5	188.6	207	212.5	215.8	199.9	183.5	189.8	184.8	180.5	187.6	165.6	177.5	151.2	145.8	145.2
	2	170.4	182.9	186.8	199.2	203.7	196.6	185.5	220.3	207.3	197.6	205.5	200.2	184.2	176.3	180.9	175.4	189.2	159.1	134.8	133
	3	179	165.1	172	185.4	182	180.1	210.8	212.9	201.3	214.6	198.3	191	180.1	186.7	183.9	179.5	182	149.6	153.2	131.8
	4		163.6	167		181.7	209.5	213.5	218.6			202.9	200.1	181.5			171.9	181.2	147.6	157.6	133
	5			171.9		188.4	185.2		200.2				202.9	201.7					155.1		
Mean Hardness		172.17	172.83	177.52	185.87	189.86	192.00	204.20	212.90	208.13	204.03	197.55	196.80	186.46	181.17	184.13	173.10	182.48	152.52	147.85	135.75
Standard Dev		6.14	9.89	10.15	13.11	9.16	11.48	12.75	7.89	7.29	9.22	9.83	5.96	8.73	5.23	3.36	5.89	4.89	4.59	9.97	6.33
180 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	167.1	185.5	196.4	198.5	197.8	196.4	223.6	211.1	210	193.9	204.9	177.5	185.4	191.1	189	166	160.4	155.7	169.2	160.2
	2	170.4	197.9	196.5	207.5	206.8	197.3	202.9	215.3	202.4	198.3	206.7	191.8	171.4	190.7	181.2	170.5	170.6	163.7	164.9	162.8
	3	179	195	193.6	210.8	215.4	208.7	208.6	213.8	215.3	202	181.7	190.6	177.9	192.1	185.5	185.5	166.6	157.6	154.5	169
	4		178.5	204.1	218		211.4	222.4	217.8	197.6		178.5	208.6	201	183.3	178.4	193.5	162.3	167.7	150.2	
	5							210.9						195					163.6		
Mean Hardness		172.17	189.23	197.65	208.70	206.67	203.45	213.68	214.50	206.33	198.07	192.95	192.13	186.14	189.30	183.53	178.88	164.98	161.66	159.70	164.00
Standard Dev		6.14	8.90	4.51	8.09	8.80	7.71	9.00	2.80	7.87	4.06	14.91	12.75	12.09	4.04	4.67	12.83	4.56	4.91	8.84	4.52
200 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	167.1	176.8	252.4	217.9	213.3	211.4	196.1	188.8	187.3	179.5	173.2	174.3	174.1	157.8	169.5	159.4	165.2	142.5	148.3	137.1
	2	170.4	189.8	233.7	229.9	209.1	219	196.2	187.1	188.6	174.3	184.5	179	178.1	187.5	165.8	154.8	164.2	148.1	146.8	135.3
	3	179	180.1	224.8	236.4	209.1	223.2	215.4	176.4	185.6	172.1	184.3	173.4	175.9	174.5	151.5	152.7	173.3	145.2	137.1	136.2
	4		185.6	250.1		224.6	208.7	190.1		185.2	176.8		179.9	170.7	171.5		166.8	158.8	152.2		
	5							215.4		188.4	189.6		161.2				170.5		145		
Mean Hardness		172.17	183.08	240.25	228.07	214.03	215.58	202.64	184.10	187.02	178.46	180.67	173.56	174.70	172.83	162.27	160.84	165.38	146.60	144.07	136.20
Standard Dev		6.14	5.77	13.24	9.39	7.32	6.70	11.91	6.72	1.57	6.81	6.47	7.47	3.13	12.19	9.51	7.64	5.98	3.71	6.08	0.90
230 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	167.1	215.3	201.7	188.8	222.4	204.1	206.8	176.2	180.1	174.1	170.7	150.8	149.1	137.6	122.4	121.2	113.8	114	121.4	117.1
	2	170.4	216.2	214.4	204.9	185.1	211.7	198.6	167.8	178.5	166.3	153.5	165.2	141.1	150.1	129.6	135.2	128.2	129.5	126.8	127.9
	3	179	211.8	202.3	217.1	195.6	182.1	186.2	166.1	183.8	165.6	150.1	166.5	141.3	151.6	123.6	120.5	133.2	131.2	126.3	128
	4		213.8	217.8				182.5	160.2			148				124.6					
	5															121.9					
Mean Hardness		172.17	214.28	209.05	203.60	201.03	199.30	193.53	167.58	180.80	168.67	155.58	160.83	143.83	146.43	124.42	125.63	125.07	124.90	124.83	124.33
Standard Dev		6.14	1.92	8.26	14.19	19.23	15.37	11.21	6.61	2.72	4.72	10.33	8.71	4.56	7.69	3.08	8.29	10.07	9.48	2.98	6.26

Table 34:Age hardened sample 8 hardness log.

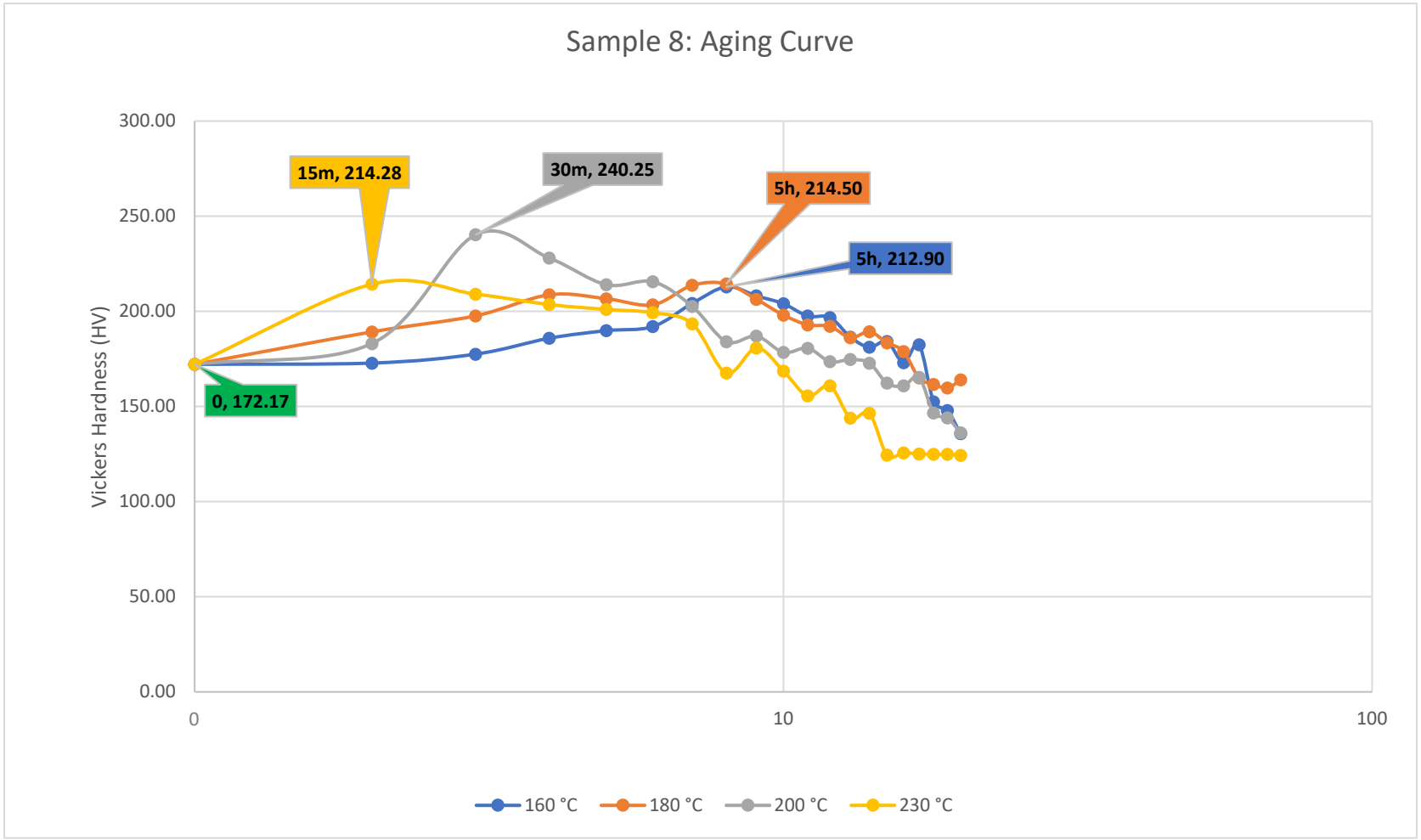


Figure 41: All plotted aging curve for sample 8.

Sample 9	sample 9	as- cast	132.7																		
160 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	142.8	118.4	139.2	129.1	143.4	120.3	149.4	161.2	169.7	178.6	156	169.8	130.1	161.5	147.8	140.7	146.1	113.8	106.9	95.2
	2	145.2	124.5	122.6	127.1	146.6	126.4	142.3	143.1	165.8	182.4	142	150.3	140.2	146.4	139	141.5	133.3	106	105	92.4
	3	126.5	119	140.8	135.3	136.2	134.5	137.9	140.4	144.4	170.7	179.9	155.3	105.3	155.7	130.5	126.1	104.2	105.9	112.6	99.5
	4	144.1	124.4		157.1	147.3				173.3	180.1				147.8		148				
	5																				
Mean Hardness		139.65	121.58	134.20	137.15	143.38	127.07	143.20	148.23	163.30	177.95	159.30	158.47	125.20	152.85	139.10	139.08	127.87	108.57	108.17	95.70
Standard Dev		8.82	3.33	10.08	13.75	5.08	7.12	5.80	11.31	12.97	5.08	19.16	10.13	17.96	7.07	8.65	9.25	21.47	4.53	3.96	3.58
180 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	142.8	133.9	146.1	131.4	163.3	139.4	163.6	158.7	156.4	166.5	142.3	159.1	135.9	171.5	153.6	134.8	142.7	147.4	127.3	125.8
	2	145.2	132.3	128.7	138.4	162.2	155.2	173.2	158	151.5	153.2	164.6	159.1	139.3	161.8	153	147.1	153.1	157.4	134.3	125
	3	126.5	114.2	132	157.1	136.1	153.4	173.4	157.5	178.1	150.2	155.5	141.1	179.2	172.2	147	162.3	156.4	159	123.8	131.3
	4	144.1	128.3			136.1	154.1	155.9	151.5	182.4						144					
	5															146.4					
Mean Hardness		139.65	127.18	135.60	142.30	149.43	150.53	166.53	156.43	167.10	156.63	154.13	153.10	151.47	168.50	148.80	148.07	150.73	154.60	128.47	127.37
Standard Dev		8.82	8.96	4.00	13.29	15.39	7.45	8.43	3.32	15.42	8.68	11.21	10.39	24.08	5.81	4.26	13.78	7.15	6.29	5.35	3.43
200 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	142.8	147.8	147.8	153.6	152	166.4	165	166.9	149.8	145.7	156.3	155.9	154	131.6	137.5	141.6	125.4	131.8	120.5	104.4
	2	145.2	143.1	145.5	140.1	142.6	153.7	159	162.9	167.8	149.6	163.3	150.8	154.2	147.7	147	135.4	126.1	131.7	114	133.5
	3	126.5	148.3	143.1	161.3	162.5	159.4	167.7	185.6	139.5	158	163	147.8	145	150.7	135.6	124.5	121.2	134.1	131.5	136.2
	4	144.1	131	146			161.6	170.8												137.6	
	5							179.2													
Mean Hardness		139.65	142.55	145.60	151.67	152.37	160.28	168.34	171.80	152.37	151.10	160.87	151.50	151.07	143.33	140.03	133.83	124.23	132.53	125.90	124.70
Standard Dev		8.82	8.05	1.94	10.73	9.96	5.27	7.46	12.12	14.32	6.29	3.96	4.10	5.25	10.27	6.11	8.66	2.65	1.36	10.63	17.63
230 °C	Time	0	15m	30m	1H	1H30m	2h	3h	5h	10h	12h	15h	18h	1d	1d12h	2d	2d12h	3d	4d	5d	6d
Reading	1	142.8	138.5	144.7	153.6	152.1	157.5	160.7	165.8	157.9	154.3	141.1	153.4	144	129.7	145.5	111.1	126.2	106.5	115.4	112.9
	2	145.2	131.9	147.4	140.1	131.9	150.5	165.3	155.8	130.9	134.8	142.4	139.3	136.5	147	123.5	124	124.1	126	118.4	115.7
	3	126.5	115.6	147.7	161.3	162.5	166.7	162.8	146.7	135.2	132.2	153.8	145	138.3	148.3	102	111	124.7	124.5	112.1	115.5
	4	144.1								165.3	158.7	136.9		140.3					104.1		117.6
	5																				
Mean Hardness		139.65	128.67	146.60	151.67	148.83	158.23	162.93	156.10	147.33	145.00	143.55	145.90	139.78	141.67	123.67	115.37	125.00	115.28	115.30	115.43
Standard Dev		8.82	11.79	1.65	10.73	15.56	8.12	2.30	9.55	16.85	13.44	7.23	7.09	3.22	10.38	21.75	7.48	1.08	11.58	3.15	1.93

Table 35: Age hardened sample 9 hardness log.

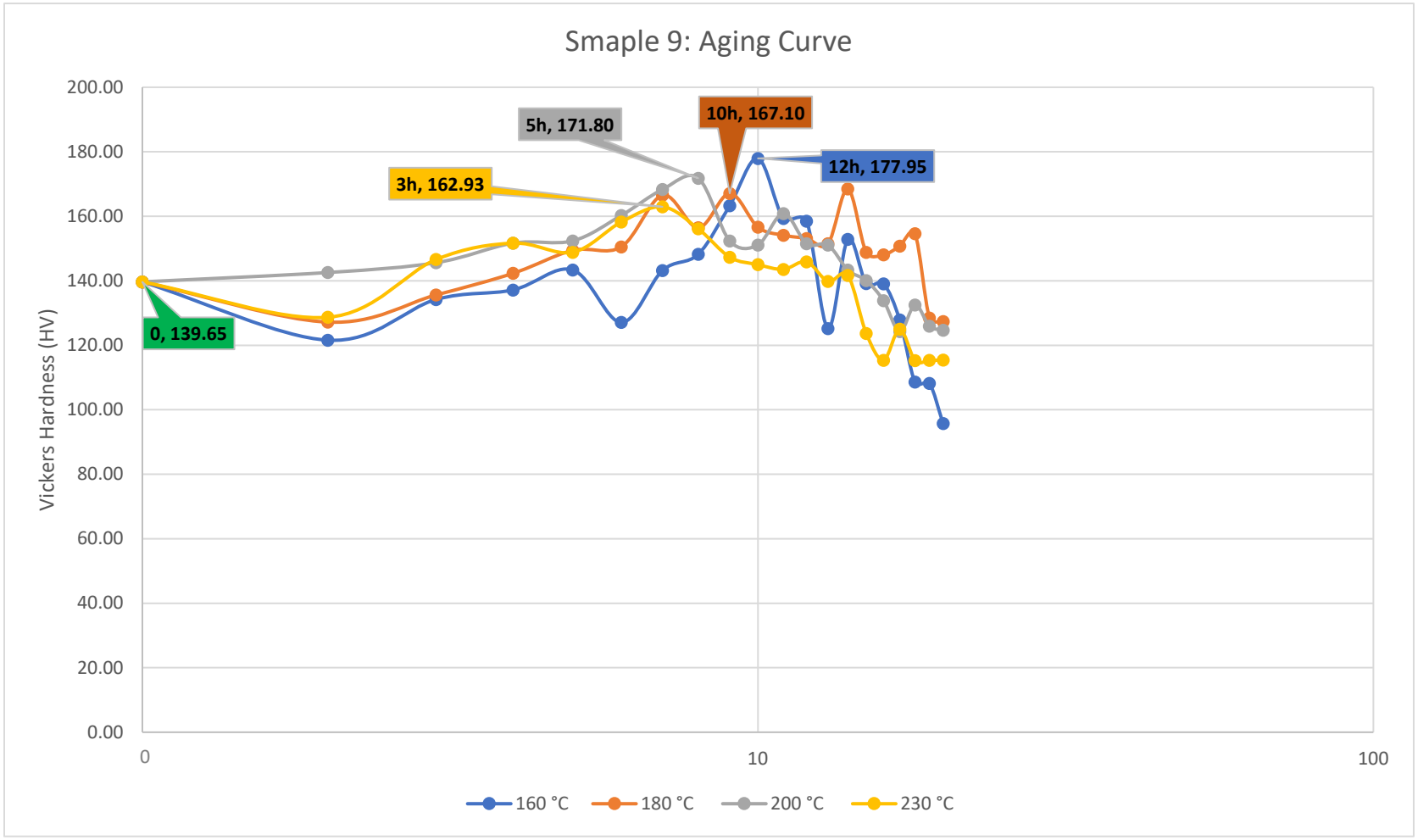


Figure 42: All plotted aging curve for sample 9.

Factor Levels	As cast	Solution treated	Aging
0.00	94.94	107.34	152.51
1.00	78.89	99.42	146.45
2.00	88.48	113.35	155.53
3.00	126.25	144.52	204.85

Silicon

Factor Levels	As cast	Solution treated	Aging
0.00	60.77	86.30	88.94
1.00	102.59	118.59	163.65
2.00	108.40	133.27	190.81
3.00	101.68	108.31	188.72

Copper

Factor Levels	As cast	Solution treated	Aging
0.00	55.79	58.78	83.02
1.00	97.23	116.51	181.21
2.00	121.64	139.91	201.77
3.00	98.77	131.28	166.12

Magnesium

Factor Levels	As cast	Solution treated	Aging
0.00	80.82	96.19	145.60
1.00	83.50	85.82	149.61
2.00	108.11	140.58	170.37
3.00	101.00	123.87	166.54

Zinc

Factor Levels	As cast	Solution treated	Aging
0.00	88.59	99.33	157.52
1.00	118.99	133.37	173.75
2.00	81.36	91.22	134.55
3.00	84.48	122.56	166.29

Manganese

Factor Levels	As cast	Solution treated	Aging
0.00	88.75	111.16	141.60
1.00	103.26	125.25	180.29
2.00	90.76	106.29	160.93
3.00	90.67	103.77	149.29

Tin

Factor Levels	As cast	Solution treated	Aging
0.00	85.00	84.83	122.35
1.00	91.97	125.04	187.36
2.00	104.11	120.64	161.55
3.00	92.35	115.95	160.86

Chromium

Factor Levels	As cast	Solution treated	Aging
0.00	79.03	119.16	163.53
1.00	92.75	108.91	165.98
2.00	76.34	75.49	106.50
3.00	125.32	142.91	196.11

Nickel

Table 36: Compilation of data logs for figure 30 to 37 in orthogonal array analysis.